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# INVESTIGATION INTO MITIGATION OF ALKALI-SILICA REACTION USING SELECTED SCMs IN PRESENCE OF POTASSIUM ACETATE DEICER

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# INVESTIGATION INTO MITIGATION OF ALKALI-SILICA REACTION USING SELECTED SCMs IN PRESENCE OF POTASSIUM ACETATE DEICER

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ABSTRACT

Alkali-silica reaction (ASR) is one of the most recognized durability problems in concrete leading to premature deterioration of different types of concrete structures. Recent investigation into premature deterioration of airfield concrete pavements indicated the aggressive effects of alkali-acetate and alkali-formate based deicers on concrete containing marginal aggregates, in particular on their potential to induce ASR. This dissertation presents the results and analysis from a research study conducted to evaluate the effectiveness of selected supplementary cementitious materials-SCMs (fly ash and slag) as ASR mitigation measures in presence of potassium acetate based deicer.

Five aggregates that encompass a range of mineralogies and reactivity were studied in combination with three fly ashes with substantially different chemical compositions, and a Grade 120 slag. These supplementary cementing materials (SCMs) were evaluated at different cement replacement levels: fly ash at 15%, 25% and 35%; and slag at 40% and 50%, using standard and modified ASTM C 1567 mortar bar tests, along with standard and modified ASTM C 1260 (mortar bar) tests and modified ASTM C 1293 (concrete prism) tests. Subsequent to the initial investigation with the three fly ashes, extensive investigation was conducted to evaluate the influence of chemical composition of fly ash on mitigating deicer-induced ASR, using twelve fly ashes and one reactive aggregate (Spratt limestone). The twelve fly ashes represented the low lime-, intermediate lime- and high lime-fly ash categories. In these investigations, all the fly ashes were used at a cement replacement level of 25%.

In addition to expansion measurement on test specimens, changes in dynamic modulus of elasticity and microstructure of the mortar and concrete samples exposed to deicer solutions was investigated. Also, changes in pH of the deicer solutions were monitored. In addition to determining the bulk chemical composition of fly ashes, X-ray diffraction studies (XRD) were conducted to characterize the crystalline compounds present in the fly ashes. The role of the various chemical constituents of the fly ash and their correlation with the expansions of the mortar bars was explored by conducting regression analyses.

In general, the chemical composition of fly ash, particularly the lime (CaO) and sulfate ($SO_4$) levels, played a significant role in determining the effectiveness of fly ashes in mitigating ASR induced by potassium acetate exposure. Low lime and intermediate lime fly ashes performed significantly better than the high lime fly ash at 25% and 35% cement replacement levels. High lime fly ashes showed a negative interaction in the presence of potassium acetate deicer and were ineffective in controlling ASR at all levels of dosages considered in the study. Slag at 50% cement replacement level was more effective in mitigating expansions compared to 40% dosage level. Besides the dosage and type of fly ash and slag, their effectiveness was also dependant on the type of aggregate.

# DEDICATION

I would like to dedicate this dissertation to my mother-Kalpana Desai, my father-Bipinchandra Desai, my younger sister-Rachana Desai and my loving wife Tanvi. I cannot imagine myself at this stage of my life without their support, blessings and good wishes. These people have inspired me to work hard, be persistent and be a good human being along with being a professional.

## ACKNOWLEDGEMENTS

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*Matru Devo Bhava-* Respect your mother like a God

*Pitru Devo Bhava-* Respect your father like a God

*Acharya Devo Bhava-*Respect your teacher/mentor like a God

I believe in these values and am thankful to my mother, my father and everyone who has been a teacher to me in some form or other and taught me things that have given me the ability to reason and have a rational approach towards life. First of all I would thank Dr.Prasad Rangaraju for being my advisor and being my teacher during my interaction with him over the past three years. His guidance and support were always there for me when I faced any stumbling block in my work. His understanding nature, dedication towards his work and passion for research has always inspired me. I wish I can emulate his hard working and untiring attitude towards work.

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# CHAPTER I INTRODUCTION

## 1.1 General

Alkali-silica reaction (ASR) is one of the most recognized durability problems in concrete leading to premature deterioration of different types of concrete structures. It is a chemical reaction between certain reactive siliceous minerals present in siliceous aggregates and the alkali hydroxides of the pore solution of concrete and portlandite of the hydrated cement paste, resulting in a gel like reaction product, which is volumetrically unstable, called ASR gel (Touma et. Al 2001, Folliard et al. 2003). The production of gel in itself is not harmful, but the gel being hygroscopic in nature absorbs moisture and expands to a volume greater than that of reacted materials exerting internal pressure which ultimately leads to the cracking of concrete. Map cracking on the surface of concrete is the most common visual evidence of the occurrence of ASR. The severity of this problem can be judged by its occurrence, not just in the United States, but through out the world. Numerous scientific papers have been published on this issue and its related mitigation since its initial discovery by Stanton in the late 1930s and a body of knowledge has been developed on this durability problem and its mitigation (Hobbs 1982, Swamy et al. 1992, Folliard et al. 2003). However, the basic mechanisms of this distress are still debated with a consensus on few fundamental factors.

Recent observations on some prematurely distressed airfield concrete pavements by the Federal Aviation Administration (FAA) have suggested that the frequent use of a new generation of alkali-acetate and alkali-formate based airfield deicing and anti-icing chemicals may have a role to play in these distresses. This led to an investigation into the potential of these deicers in initiating and accelerating ASR. The results of this study concluded that these deicers, mainly sodium and potassium salts of acetate and formate, have a significant potential in having a detrimental effect on the concretes exposed to them by causing deleterious ASR in mortar and concrete specimens containing reactive aggregates. However, the deleterious ASR symptoms were not seen in specimens containing non reactive aggregate (Rangaraju et al. 2007)

The mitigation of ASR under the traditional exposure conditions, where the source of alkalis is mainly internal, has been well documented and the commonly employed mitigation measures include using one or all of the following: using non-reactive aggregates, using low alkali cement, using supplementary cementitious materials (SCMs) like fly ash, slag, silica fume among others; and using lithium based chemical admixtures. However, the new generation acetate and formate based deicers pose a different situation and hence a different interaction mechanism between them and the concrete making materials. This study is an attempt to understand the mechanisms and potential of SCMs like fly ash and slag to mitigate or aggravate the ASR like deleterious reactions in concrete in the presence of non-chloride based deicing chemicals.

## 1.2 Problem Statement and Research Significance

Deicers and anti-icers are a necessity during the winter weather conditions to aid snow removal and prevent ice formation on the pavements to create safe driving conditions. Over the last decade new deicers such as acetate and formate based deicers have replaced the traditional deicers and have gained popularity because of its environmental friendly characteristics and better effectiveness in deicing/anti-icing.

With the increase in the use of these deicers on airfield concrete pavements, an increasing number of airports have reported premature deterioration of their pavements. Laboratory investigations have also confirmed the deleterious effects of these deicers (IPRF 2007).

Among the mitigation alternatives used for preventing concrete pavements from the damaging effects of these alkali-acetate and alkali-formate based deicers, the best alternative would be to stop or limit the use of these deicers. However, the availability of a suitable alternative deicer with no such damaging effects, while having the same or better deicing and anti-icing properties and eco-friendliness as these deicers, is not present at this point in time.

Another ideal mitigation alternative would be to use non-reactive aggregates, thereby removing the source of reactive silica which is essential for ASR to propagate and hence, minimize the potential of ASR in concrete pavements. This alternative has been tested as a part of FAA project 03-9 and it was found that no ASR-related effects were observed in the concrete and mortar specimens, containing non-reactive aggregates, exposed to the acetate and formate deicers. However, the availability of non-reactive aggregates in most parts of the United States is scarce and hauling them to the project site may prove to be economically restraining.

Using low alkali cements and limiting the total alkali content of concrete have been suggested to be effective in reducing the ASR potential. However, this may not be effective in situations where the source of alkalis is external (like deicers). This alternative has been tested and studied by Rangaraju et al. by exposing mortar and concrete specimens, made using high and low alkali cements, to acetate and formate based deicers and it was found that the low alkali cements had only delayed the expansions of the specimens (Rangaraju et al.

2007) and hence, using low alkali cement alone might not be an ideal solution against ASR damage caused by these deicers.

Traditionally, mineral admixtures or supplementary cementitious materials (SCMs) have been used in concrete mix proportions to effectively mitigate or eliminate ASR. SCMs like fly ash, ground granulated blast furnace slag (GGBFS) and silica fume are some of the most commonly used SCMs and in the last few decades the use of chemical admixtures like lithium based compounds have proven to be very effective against ASR.

Much of the research done so far was focused on situations where the alkalis are contributed by the internal sources of concrete, mostly Portland cement, SCMs and aggregates among others. In this regard, there is dearth of information in the literature on the effectiveness of SCMs in mitigating ASR in concrete induced by the new generation of pavement deicing chemicals.  Due to the lack of options to choose alternate deicers having the same effectiveness and environmental friendliness, an immediate discontinuation of the use of these deicers doesn't seem to be a viable option and its use is likely to grow. Hence, it becomes imperative to make the concrete, being used for new construction of pavements and structures, with materials that make it resistant to the exposure of these deicers and prevent premature failures. SCMs have been effective against ASR due to internal alkali sources of concrete and also provide many other benefits to the concrete, but their ASR mitigation potential and reaction mechanisms against the exposure to alkali-acetate and alkali-formate based deicers needs to be investigated.

This research study is an attempt to investigate the effectiveness of selected SCMs in mitigating ASR induced by potassium acetate deicer, which is the most widely used airfield pavement deicer in the United States, and provide recommendations for using them in new concrete construction exposed to deicers.

## 1.3 Objectives

The principal objectives of this study are:

1. To study and determine the potential of selected supplementary cementitious materials (fly ash and ground granulated blast furnace slag) to mitigate ASR induced by potassium acetate deicer.
2. To study the effect of dosage and chemical composition of fly ashes and slag to mitigate deicer induced ASR.
3. To study the mechanisms involved in the interaction of fly ash and slag, Portland cement and aggregates with potassium acetate deicer.
4. Develop recommendations on the use of fly ash and ground granulated blast furnace slag in new concrete construction and suggest a test procedure to evaluate fly ash and slag for deicer induced ASR mitigation.

## 1.4 Scope of Research

This research study is limited to the use of selected materials and various standard and modified test methods to address the objectives listed in section 1.3. The selected materials include SCMs (fly ash and slag), portland cement, aggregates and potassium acetate deicer.

Fifteen commercially available fly ashes from power plants across the United States and having a wide range of chemical compositions (lime content in specific) were used in this study. One commercially available ground granulated blast furnace slag (GGBFS), by product of iron production, was used as the other selected SCM. For the sake of simplicity from here on GGBFS will be referred to as slag. Slag being more consistent in its chemical composition across the sources, only one slag was used. Type-I high alkali portland cement with an $Na_2Oeq.$ of 0.82% was used through out the research study.

Six aggregates were used in this study with five being from different sources across the United States and one from Canada. These aggregates were selected based on their established field history of being alkali-silica reactive or non-reactive and were classified into three categories with two aggregates of each category. The three categories were: 1) highly reactive to reactive 2) moderately to slowly reactive and, 3) non reactive. The non-reactive aggregates were used as reference and were used as reference aggregates.

A commercially available potassium acetate deicer (50% wt. solution) was used for the entire study to investigate the effectiveness of fly ashes and slag as deicer induced ASR mitigation alternatives. In the absence of standard test protocols to evaluate SCMs exposed to deicers, modifications were made to standard tests (ASTM C 1260 for ASTM C 1567 for mortar and ASTM C 1293 for concrete) and a series of tests were conducted. Mortar bars and concrete prisms were made using the six different aggregates and the SCMs (fly ash and slag) were used at different cement replacement levels. The standard tests involved soaking the samples in 1 normal (1N) sodium hydroxide solution, while the modified versions of the standard tests required the soaking of samples in potassium acetate deicer solution. The results of the standard tests were used as a reference for comparing them to the modified tests. Complementary studies such as scanning electron microscopy (SEM), energy dispersive X-ray (EDX) analyses were conducted to study the microstructure of the test specimens, while dynamic modulus of the mortar and concrete specimens was measured to study the influence of soak solutions (potassium acetate deicer and 1N NaOH solutions) on the physical deterioration of the samples during the test regimen. pH studies and silica dissolution studies using inductively coupled plasma (ICP) technique were conducted to study the reaction mechanisms of the potassium acetate deicer with the SCMs and aggregates.

The details of the all the above mentioned tests are explained in chapter 3 of this dissertation. In sum, the scope of this research was limited to testing 6 different reactive aggregates, 15 fly ashes with a wide range of chemical composition, 1 slag, and high alkali cement using different standard and modified test protocols to achieve the defined objectives.

The experimental program involved conducting a total of 124 mortar bar tests and 32 concrete prism tests. Of the 124 mortar bar tests, 62 were standard tests and the other half were modified tests. All the concrete prism tests were the modified tests.

## 1.5 Research Approach

- Conduct literature review of past studies on ASR mitigation alternatives in general and for ASR induced by deicers, with an emphasis on ASR mitigation using fly ash and slag.
- Select aggregates which represent a range of reactivity based on their established field history of being alkali-silica reactive or non-reactive.
- Collect commercially fly ashes that represent a range of chemical composition, specifically the lime content, and a commercially available slag.

- Study test procedures to evaluate aggregates for potential alkali-silica reactivity and effectiveness of SCMs against ASR.
- Develop an experimental program with tests to study the influence of SCMs in mitigating ASR due to potassium acetate deicer.
- Conduct complementary studies to understand the mechanisms of the reactions in the standard and modified ASTM C 1260, ASTM C 1567, ASTM C 1293 tests.
- Provide recommendations for using fly ash and slag as ASR mitigation alternatives in new concrete construction to be exposed to potassium acetate deicer. Suggest test procedure to evaluate aggregates and SCMs (fly ash and slag) for deicer induced ASR mitigation.

## 1.6 Organization of the Dissertation

This dissertation is written in five chapters, each chapter evolving into the other and complementing each other. Chapter 1 is an introduction to this research study and states the significance and need for this research. It also defines the principal objectives and scope of this study, followed by the approach being used in conducting this research.

Chapter 2 is a review of the literature on past studies and state-of-the art on the ASR durability issue in general, its mechanisms, traditional and non-traditional ASR mitigation alternatives with an emphasis on the role of SCMs-fly ash and slag in particular, in ASR mitigation. It also presents a review of the research related to alkali-acetate and alkali-formate deicers causing ASR and its mitigation. This chapter also discusses the various test methods employed in this study and their pros and cons. Previous research related to the materials selected for this study is also discussed in this chapter.

Chapter 3 discusses the materials and test methods (standard and modified) used in this study and lays out an experimental program.

Chapter 4 presents the various results obtained by executing the experimental program presented in chapter 3. This chapter also presents the analysis and discussion of the results.

Chapter 5 concludes this dissertation by stating the principal findings from this study and draws conclusions to the principal objectives laid out in chapter 1. Based on the principal findings, recommendations for putting our research into practical use and scope for further research are provided for the benefit of the reader.

# CHAPTER II LITERATURE REVIEW

## 2.1 General

This chapter deals with literature related to the basics of alkali-silica reaction (ASR) and its occurrence, the theories related to the basic reaction mechanisms of ASR, the factors influencing ASR, and the commonly used ASR mitigation measures. This chapter also gives a background of the commonly used deicing chemicals on concrete pavements and research related to their effects on the durability of concrete with specific focus on the potential of deicers in causing ASR. The research related to the influence of alkali-acetate and alkali-formate based deicers on concrete pavements is fairly recent and limited. However, this chapter cites this limited research and its findings along with the various modified test methods used to evaluate aggregates for their ASR potential and materials used as mitigation measures when exposed to the deicers.

## 2.2 Alkali-Silica Reaction

Alkali-Silica Reaction is a distress in concrete that is caused by a reaction between the available alkalis from the cement paste, and/or other external alkali sources, and certain reactive forms of silica within an aggregate. ASR was identified in many large structures, including large dams, ship locks, parking houses, main roads, pavements and other concrete structures, all over the world since its initial discovery by Stanton in 1940, in highway structures in California. ASR is the deleterious expansion reaction between certain reactive components of the aggregate and alkalis from the cement paste leading to the formation of an alkali-silica gel which expands and causes cracking in concrete. The amount of gel and the swelling pressures are variable and are dependent on various factors like amount of reactive materials, availability of alkalis from internal and external sources, temperature, gel composition and availability of moisture. Typical ASR distress features in concrete structures are that of closed spaced polygonal cracking called 'Map Cracking', spalling of concrete surface as 'pop-outs', expansion leading cracks and consequent misalignment of structural elements, and extrusion of gel through the cracks or its presence in the fractures and/or aggregate particles (Poole 1992).

### 2.2.1 Factors Influencing ASR

Though the basic mechanisms of the alkali-silica reaction and its mitigation are still debated, the factors influencing ASR are widely accepted and it is the combination of these factors that determine the rate of deterioration of the concrete structure. The three important factors for ASR to occur are as follows:

1. Sufficient Alkalis (contained in the pore solution),
2. Reactive silica (poorly crystallized silica present in certain aggregates) and,
3. Sufficient moisture.

*1) Sufficient Alkalis*

Alkali content of cement is one of the main factors driving the ASR reaction.

While limiting the alkali content of cement is one way to ensure that the ASR effects are minimized, this measure by itself is not adequate to address ASR, particularly when alkalis from external sources can penetrate concrete and potentially trigger deleterious reactions. The alkalis that trigger ASR in concrete can come from any of the following sources:

- Portland Cement:

Cement is main contributor of alkalis in concrete and they are in the form of $Na_2O$ and $K_2O$. As per ASTM C 150, low alkali cements have a $Na_2O$ equivalent of less than 0.6% and for high alkali cements this may be up to 1.1%.

- Supplementary Cementing Materials (SCMs):

Limits are placed on the alkali content of SCMs (like fly ash, silica fume and slag) as per ASTM C 618 and ASTM C 311, but most agencies do not consider the contribution of alkalis from the SCMs towards calculating the total alkali content on concrete (Folliard et al. 2003). Opinions are divided over the contribution of the alkalis present in fly ash and slag towards accounting for the total alkali content of the concrete and hence its involvement in the alkali-silica reaction.

- Certain types of volcanic aggregates (esp. basalts and volcanic glass)
- Chemical admixtures
- Alkaline Soils
- External sources (seawater and deicing salts)

These salts are a common source for external alkalis on highways, airfield pavements and structures exposed to sea water.

*2) Reactive Silica*

Aggregates from rocks are termed to be 'reactive' depending on the particular form of silica (crystalline or amorphous) present in its structure. Though most of the aggregates used in the USA are siliceous in composition, i.e. high silica ($SiO_2$) content, they are not necessarily reactive (Sarkar et al. 2004, Swamy 1992). Certain reactive aggregates do not exhibit maximum expansion unless the aggregate is present in a critical range. This critical range or proportion of reactive aggregates required for the maximum expansion to occur is called the 'pessimum proportion'. Besides this, it also depends on the exposure conditions which may or may not be conducive for the reactive aggregates to trigger an expansive reaction leading to damage and durability issues. The difference in expansion of different potentially reactive aggregates mainly depends on the following:

(i) the inherent reactivity of their constituent mineral phases or rock types,

(ii) grain size of the reactive particle, and

(iii) the proportion of these reactive phases within the reactive aggregate.

Table 2.1 shows some of the potentially reactive rocks and minerals which are known to cause ASR. Several of the rocks listed (for e.g. granite gneiss and some quartz formations are slowly reactive and may take up to 20 years to show signs of deleterious ASR reactivity (CSA A23.1 2000)). Ferraris (Ferraris 1995) has elaborately discussed the most common alkali-silica reactive rocks and their distress characteristics when they are affected by ASR. A schematic showing the texture, morphology and compositions of primary alkali susceptible rocks and characteristic patterns when affected by ASR is shown in figure 2.1

Table 2.1 List of Reactive Silica Minerals and Rocks

| | |
|---|---|
| Andesites, | Opal, |
| Argillites, | Opaline Shales |
| Certain Siliceous Limestones & Dolomites, | Phylites, |
| Chalcedonic Cherts, | Quartzites |
| Chalcedony | Quartzoses |
| Cristobalite | Cherts, |
| Dacites | Flint |
| Glassy or Cryptocrystalline volcanics | Rhyolites, |
| Foliated Gneiss | Schists |
| Granite Gneiss | Siliceous Shales, |
| Graywackes, | Strained quartz |
| Metagraywackes | Other forms of quartz, |
| Siltstones | Synthetic and Natural siliceous glass |
| | Tridymite |

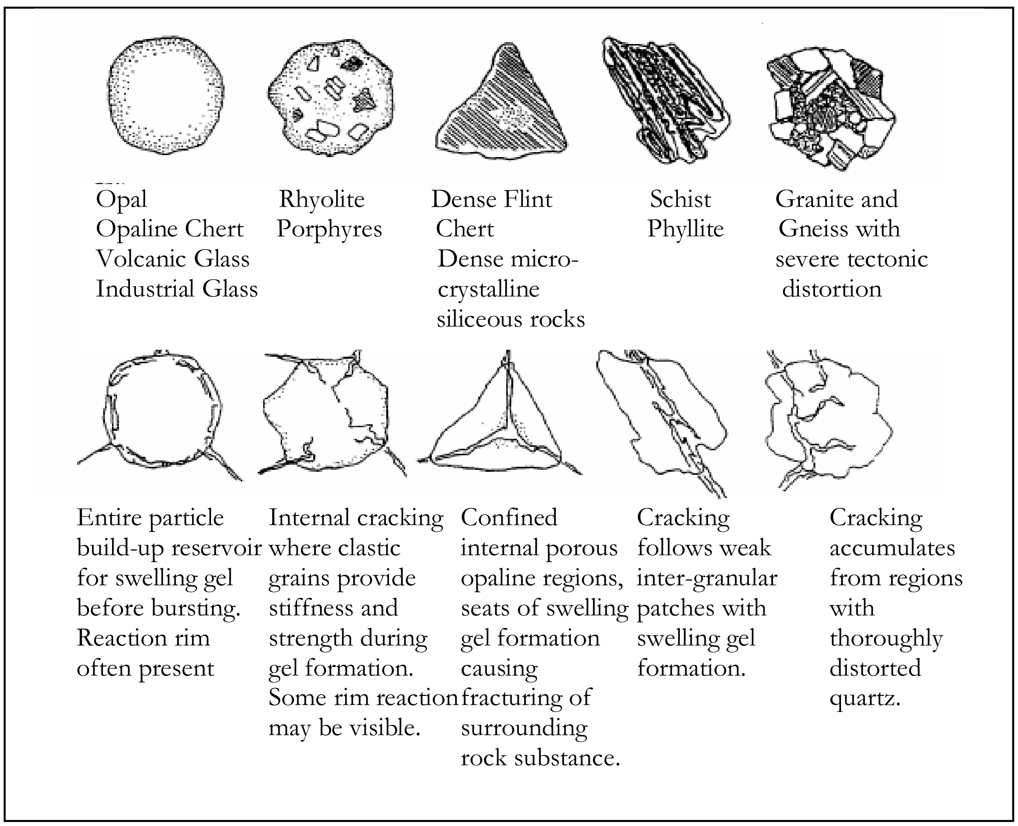



Figure 2.1 Schematic Showing Texture, Morphology and Compositions of Primary Alkali Susceptible Rocks and Characteristic Patterns when Affected by ASR

*3) Sufficient Moisture*

Moisture acts as a medium for migration of alkali ions to the reactive sites once the aggregate structure is broken down by the reaction. With the advancement of the reaction, moisture acts as a transport medium for taking the ions to the reactive silica through the cracks developed due to the tensile stresses generated. The wetting and drying cycles of an exposed concrete surface facilitates the migration of alkalis and also increases the concentration of salts, especially in cases of deicing salt application and sea water exposure. According to a study by Chatterji, a relative humidity (RH) of at least 80% is essential for ASR to propagate (Chatterji 2005). At RH of 80% or higher the ASR gel, formed as a reaction product which is hygroscopic in nature, expands and causes further tensile stresses.

### 2.2.2 Reaction Mechanisms

The ASR reaction mechanism can be divided into two stages (Sarkar et al. 2004), the chemical reaction and the distress mechanism that follows the chemical reaction. The alkali-silica reaction is basically a dissolution reaction where the reactive silica dissolutes in the high pH environment generated by the alkali hydroxides in the pore solution of concrete. Chatterji found that the pore solution containing $Ca^{+2}$, $K^{+}$, $Na^{+}$, $OH^{-}$ and $SO_4^{--2}$ ions changes drastically in its chemical composition after a short span of 8 hours. The $OH^{-}$ ion and alkali ion concentration increased rapidly while the $Ca^{+2}$ and $SO_4^{-2}$ ions were left to a trace in the pore liquid. As a result of this, the pH of the solution increases fostering the ASR (Chatterji 2005).

The pH of the alkali solution in the micro pores of the hardened concrete matrix having the dissolved alkali hydroxides is highly basic, i.e. ≥12.5 (Fournier et al. 2000). In this highly alkaline solution, the hydroxyl ions attack the Si-O-Si layer near the surface of the reactive siliceous aggregate and break it down. According to different studies, this reaction takes place on the surface of the aggregate if the silica is well crystallized and on the inside, if the silica has an amorphous structure or is poorly crystallized. This layer is converted to Si-O- after the breakdown which attracts $Na^+$ and $K^+$ alkali ions to maintain charge equilibrium. The resultant product of this reaction is a gel essentially of silica, alkali (Na+ or K+), calcium and water. When the gel formed is rich in calcium, it is non expansive (Chatterji 2005, Sarkar et al. 2004, Folliard et al. 2003, Fournier et al.2000, Ferraris 1995). The following equation describes the process of breaking down of silica of the aggregates:

**Equation 1:** $\equiv Si\text{-}OH + OH^- \rightarrow \equiv Si\text{-}O^- + H_2O$

**Equation 2:** $\equiv Si\text{-}O\text{-}Si\equiv + 2OH^- \rightarrow 2(\equiv Si\text{-}O^-) + H_2O$

The reaction process can be viewed as a two step process:

*Step 1:*

Silica + Alkali = Alkali-Silica-gel (Sodium Silicate)

$SiO_2 + 2NaOH + H_2O = Na_2SiO_3.2H_2O$ (2KOH can replace 2 NaOH)

*Step 2:*

Gel Reaction Product + water = Expansion

The reaction products of this reaction lead to the development of pressure within the concrete and when this pressure exceeds the tensile strength of concrete, it leads to the formation of micro and macro cracking.

## 2.3 Deicing Chemicals and its Effects on ASR in Concrete

Chemicals used during the winter operations mainly perform either of the two functions, i.e. to melt the snow and ice (Deicing) or to prevent them from bonding to the surface of the pavement (Anti-icing) and the chemicals that perform these functions are called 'Deicers' and 'Anti-icers' respectively. These are available in solid form (e.g. sodium chloride, sodium acetate, sodium formate) or liquid form (calcium chloride, potassium acetate, aqueous solutions of glycols and urea). Some chemicals are used interchangeably as deicers or anti-icers, e.g. Potassium acetate and calcium chloride (Krichner 2001).

Deicers can be classified based on their chemical composition. Most of the highway deicers are chloride based. Though some acetate based deicers are also used for highways, their use is limited when compared to the chloride based deicers. The airfield deicers traditionally and presently used are mostly acetate, glycol or formate based. For the sake of reporting the literature on deicers, they have been classified into 'Traditional' and 'alkali-acetate and alkali-formate' based deicers.

### 2.3.1 Traditional Deicers

#### Chloride Based Deicers

These are mostly used in highway deicing and anti-icing operations. The main reason for their widespread use is their effectiveness in snow melting at a low cost. The most common chloride based deicers are sodium chloride (NaCl or Rock Salt) and calcium chloride. The consumption of salt as a deicer alone is more than 15 million tons a year (Krichner 2001). Other chloride based deicers include, potassium chloride, magnesium chloride and blends of the above mentioned deicers. The solid deicers are used in combination with abrasives like cinders or sand so as to increase the traction of vehicles during the snowfall. The problem with using solid deicers is their ability to stay back on the surface during snow storms and hence their application needs to be frequent (Salt Institute 2004).

Considerable research has been done to understand the influence of these commonly used deicers, sodium and calcium chloride, in initiating or aggravating ASR in concrete. Kawamura et al.(Kawamura et al.1990, Kawamura et al.1994), Duschene et al. (Duschene et al.1996, Duschene et al.2003) have explained the corrosion potential of these deicers and this is supported by case studies cited by other researchers on the corrosion of embedded steel in highway concrete pavements exposed to sodium and chloride salts. Lee et al. (Lee et al.1997) studied various chloride based deicers and found that they have the potential to aggravate ASR in concrete. The mechanism behind this was believed to be the increase in the pH of the pore solution of concrete when the sodium and calcium chloride deicers react with the hydration products of concrete liberating OH- ions. This high alkaline environment in the presence of reactive silica and moisture from the surrounding pore solution fosters ASR. Wang et al. (Wang et al. 2005) investigated the effects of five deicing chemicals including sodium and calcium chloride (with and without corrosion inhibitors), potassium acetate and an agricultural deicing product. Results of their investigation concluded that the calcium chloride deicers had the most damaging effect on concrete and mortar samples when exposed to freeze-thaw and wetting-drying conditions. Sodium chloride was less damaging to concrete compared to calcium chloride, but caused more damage compared to potassium acetate and agricultural deicer.

The common conclusions of the studies on chloride based deicers and their detrimental effects on concrete and mortar were associated with the salt crystallization and precipitation, leaching of the cement hydration products (CH and ettringite), a chemical reaction between the deicing chemicals and concrete materials (Ca–Al–Cl–S hydrate formation –Wang et al. 2005) and increase in the pH resulting of pore solutions

Magnesium Based Deicers

Besides the magnesium chloride deicer, there have been acetate based deicers in combination with magnesium and calcium and these have become popular because of their less corrosive effects and low environmental impact. Of these, calcium magnesium acetate (CMA) is more popular because of its low biological oxygen demand (BOD). These deicers are mostly used on regular pavements and not on airfield pavements due to their ineffectiveness at very low temperatures. But CMA has its drawbacks in the form of high cost and potentially harmful to concrete. Lee et al. (Lee et al. 1997) and Cody et al. (Cody et al. 1996) conducted a series of experiments with NaCl, CaCl2, $MgCl_2$, Magnesium acetate and CMA by exposing concrete specimens to wet/dry and freeze thaw conditions and their results indicated that CMA had the most deteriorating effect on concrete by causing a de-lamination of the cement matrix. Studies by Krischner (Krischner 2001) confirmed the damaging effects of $MgCl_2$ and CMA and noted a drastic reduction in the load bearing capacity and increased scaling of concrete specimens when exposed to $MgCl_2$ and CMA.

### 2.3.2 Alkali-Acetate and Alkali-Formate Based Deicers

Due to the disadvantages of these traditional airfield deicers such as glycols and urea, Federal Aviation Administration (FAA) advocated the use of alternative deicers which have been widely used in Europe. These deicers with new formulations were based on potassium acetate, sodium acetate, sodium formate and potassium formate. These had the functional benefits of being effective in deicing and anti-icing at low temperatures and, the environmental benefits in terms of low BOD. However, not much information is available on their potential side effects related to corrosion or ASR in concrete. Wang et al. (Wang et al. 2005) used potassium acetate, an agricultural deicing chemical and three chloride based deicers to study their effects on concrete and mortar samples when exposed to freeze-thaw and wetting-drying conditions. Their study indicated that chloride deicers had the most damaging effect, while potassium acetate exposed samples had minor scaling with no significant mass loss or signs of cracking on the samples. However, this study was not focused on investigating the effects of potassium acetate in causing ASR in concrete and mortar. A detailed study with the new generation alkali-acetate and alkali-formate deicers, with respect to their potential to initiate or accelerate ASR in concrete was conducted by Rangaraju et al. at Clemson University and the findings are complied as a report (Rangaraju et al. 2007) and is also published elsewhere(Rangaraju et al. 2005, Sompura 2006). The results of this study are mentioned in the following section (2.4).

Potassium acetate runway deicer is a 50% aqueous solution of potassium acetate by weight that includes proprietary corrosion inhibitors and indicator dye. It has been used at US airports since 1991. Of the new generation deicers; sodium acetate, sodium formate and potassium acetate are the most widely and growing airfield deicers. Among these, potassium acetate has its largest share and is expected to increase its market share in future (Rangaraju et al. 2005).

## 2.4 Past Research Related to ASR Induced by Alkali-Acetate and Alkali-Formate Deicers

The growing concerns related to the premature deterioration of airfield concrete pavements across the United States led to the initiation of a research study funded by FAA through Innovative Pavement Research Foundation (IPRF). A detailed study was conducted by Rangaraju et al. (Rangaraju et al. 2007) to evaluate the potential of alkali-acetate and alkali-formate based deicers to initiate and/or accelerate ASR. This project, IPRF 03-9, and its findings published elsewhere (Rangaraju et al. 2005, Sompura 2006) are the only sources available in literature that account for the influence of acetate and formate based deicers and its potential to initiate ASR. The results of the FAA 03-9 project provide a platform for this Ph.D. research work.

The FAA study involved testing six aggregates having a range of reactivity, two types of cement- high and low alkali, and four commonly used airfield deicers, namely sodium acetate, sodium formate, potassium acetate and potassium formate. The six aggregates used in that project are the same as those being used in the Ph.D. research work discussed in this dissertation. The details of the six aggregates are provided in Chapter 3 of this dissertation. Since no standard procedures are available to evaluate aggregates for their ASR potential when exposed to deicing chemicals, modified versions of the ASTM C 1260 and ASTM C 1293 were used along with the standard versions of the same. The modified versions of the ASTM C 1260 tests involved replacing the 1N NaOH soak solution with the deicers while keeping the testing regimen the same. While the modified version of the ASTM C 1293 test involved soaking the concrete prisms horizontally in a bath of 1N NaOH or deicing solution instead of storing them vertically in a 100% relative humidity environment. Microstructure studies were conducted on the mortar and concrete samples to understand the composition of the reaction products.

The principal findings from this study established that the alkali-acetate and alkali-formate airfield deicers not only initiated, but also accelerated the deleterious alkali-silica reactions in mortar and concrete specimens containing reactive aggregates. Specimens exposed to these deicers showed intensive cracking and expansions over the testing regime. Microstructure analysis using SEM and EDX revealed the formation of micro-cracking in the aggregate and in the cement paste-aggregate interface, ASR gel formation within the cracks and in some instances formation of dark reaction rim around the aggregate surface. A majority of the specimens exposed to deicers had similar or higher expansions compared to the standard 1N NaOH solution exposure.

## 2.5 Test Methods to Evaluate ASR

There are a number of standard test methods to evaluate the potential reactivity of aggregates and aggregate-cement combinations that have been developed over the years since its discovery in 1940 by Stanton. These methods include chemical tests, microscopic and visual examination and tests for cementitious materials-aggregate combinations in mortar or concrete. Rapid or accelerated test methods are developed so as to give conclusive results in a short period of time. However, the reliability of the accelerated test methods is questioned because of its severe exposure conditions and sometimes lack of correlations between field and laboratory tests.

Some of the tests that are more commonly used to asses the alkali-silica reactivity of aggregates include the mortar bar tests ASTM C 1260, ASTM C 1567 and ASTM C 227; and the concrete prism test ASTM C 1293. The ASTM C 1260 and ASTM C 1567 are known as the 'Accelerated Mortar Bar Tests (AMBT)' and have shown a fair amount of reliability in identifying the reactive aggregates and also the effectiveness of SCMs in mitigating ASR (Thomas et al.1999, Thomas et al. 2001, Touma et al. 2001, Malvar et al. 2001). In comparison, the other mortar bar test-ASTM C 227 test- has it drawbacks in terms of variations in results due to leaching of alkalis during the test regimen and the long length (6 months) of the test that makes it impractical to get conclusive results to make a decision on the selection of the aggregates (Rogers 1999).

ASTM C 1293 or the 'Concrete Prism Test' is considered to be the most reliable among the available test procedures to asses the potential reactivity of aggregates and the effectiveness of SCMs in mitigating ASR. There is generally a good agreement between the results of the accelerated mortar bar tests- ASTM C 1260 and ASTM C 1567, and the concrete prism test (Thomas et al.1999). In the event of contrasting results between the AMBT and the concrete prism test (ASTM C 1293); the results of the later are considered to be valid. However, the long duration of the test, 12 months for plain aggregate cement concrete and 2 years for concrete with pozzolans and slag, to conclude an aggregate to be innocuous or reactive and for SCMs to be effective in containing the deleterious expansions is a drawback. Aggregate-cementitious material combinations satisfying the acceptance criteria of both ASTM C 1260 (<0.10% expansion after 16 days) and ASTM C 1293 (<0.04% after 12 months or 24 months in case of SCMs) can be reliably used in construction.

A fair amount of work has been done by various researchers with regards to the use and development of tests to evaluate SCMs like fly ash, slag and other pozzolans to control the ASR expansions (Thomas et al.1999, Rogers 1999, Shehata et al. 2000, Duschene et al. 2000, Thomas et al. 2001, Touma et al. 2001, Detwiler 2003, Folliard et al. 2003). There have been quite a few multi-laboratory studies to validate the AMBT test, the modified version of ASTM C 1260 now known as ASTM C 1567, and the concrete prism test (ASTM C 1293). Though past research provides a substantial database for determining the efficiency of SCMs in mitigating ASR and validating various test methods for the same, these studies pertain to situations where the alkalis are contributed by cement or from other internal sources in concrete. In this regard, not much information is available in literature on the effectiveness of SCMs in mitigating ASR induced by external sources such as deicing chemicals, in particular alkali-acetate and alkali-formate deicers.

## 2.6 Past Research Related to Mitigation Measures for ASR Induced by Alkali-Acetate Deicers

This section deals with the typically used ASR mitigation measures and research related to using these alternatives in mitigating ASR induced by alkali-acetate deicer. Traditionally, the ASR mitigation measures include one or a combination of the following:

- Use of non-reactive aggregates
- Use of SCMs like fly ash, slag, silica fume and other pozzolans
- Use of lithium compounds
- Limiting alkali content of the concrete
- Use of air entrainment in concrete
- Use of physical restraints like carbon fiber wraps and steel micro-fibers.

Use of non-reactive or innocuous aggregates is an ideal solution to prevent the occurrence of deleterious ASR. However, the availability of non-reactive aggregates in all the regions of the country is not possible and hauling them to the job site from other regions might prove to be economically restraining. Research related to the use of non-reactive aggregates as an ASR mitigation alternative for concrete exposed to deicers has shown that when such aggregates are used in concrete (modified ASTM C 1293) and mortar (modified ASTM C 1260) samples the expansions are within the acceptable limits and hence confirming their mitigation ability (Rangaraju et al. 2007).

Of all the ASR mitigation measures, use of SCMs is reportedly the most successful and practical alternative. There has been a general consensus regarding the effectiveness of SCMs in suppressing the deleterious expansion due to ASR (Thomas et al.1998, Shehata et al. 2000, Duschene et al. 2000, Malvar et al. 2001, Touma et al. 2001,Thomas et al.2001, Detwiler et al. 2003, Folliard et al. 2003). While the mechanisms are not clearly understood, several possibilities have been proposed and generally accepted. However, three mechanisms are broadly accepted (Glasser 1992, Detwiler 2003):

- Dilution of the cement alkalis by the blending agents as they contain less available alkali compared to the cement they replace and liberate alkalis at smaller rates compared to cement.
- Reduced permeability and diffusivity by refining the grain size and pore size leading to discontinuous pores and decreased porosity around the aggregates. This ultimately reduces the migration of the alkalis towards the reactive aggregate particles.
- Binding of alkalis and lowering the $Ca(OH)_2$ content of the cement paste and hence lowering the pH of the pore solution. Also, the increased pozzolanic reaction makes

the SCMs react rapidly with the ions and producing C-S-H that traps the alkali ions and therefore reducing their concentration in the pore solution. The $Ca/SiO_2$ ratio of C-S-H in typical Portland cement is about 1.8 and the use of SCMs with low CaO content reduce this ratio. It is found that if the $Ca/SiO_2$ ratio is kept less than 1.5 the chances of ASR are reduced.

Fly ash has been commonly accepted as a 'must' for ASR mitigation for new concretes. Class F (low calcium, ASTM 618) fly ash at 25% cement replacements has been shown to significantly mitigate ASR. In a comprehensive study by Touma et al. (Touma et al. 2001) at the International Center for Aggregate Research (ICAR) using a range of reactive aggregates and various ASR mitigation alternatives, the following recommendations were made for ASR mitigation:

(a) 25% class F fly ash

(b) 35% class C fly ash

(c) 10% silica fume

(d) 55% slag

(e) 17% calcined clay

(f) 4.6 L LiNO3 per Kg of Na2O.

Reductions in expansions were found in mortar samples made using highly reactive New Mexico aggregates by using Class F fly ash at a minimum cement replacement of 25% while Class C fly ashes produced higher expansions (Barringer 2000). Studies by Thomas et al.(1996, 2000), Shehata et al. (2000), Duschene et al. (2001) suggest that using Class F fly ash with low lime contents at 25% cement replacement is the best alternative to mitigate ASR. High Cao content fly ash (Class C fly ash) is not advocated for use with reactive aggregates and the reason for their ineffectiveness is believed to be their inability to lower the $Ca/SiO_2$ ratio due to their high CaO content. The inefficiency of high lime fly ashes is also attributed to the more amorphous phases present in the fly ash and hence the availability of alkali for reaction is higher compared to low lime fly ashes, where the alkalis may be bound in the crystalline phases (Shehata and Thomas 1999).

Slag offers similar advantages to class F fly ash, but only when used in higher quantities. Slag of grades 100 and 120 are preferred to grade 80 and slag with low lime content is advocated. High levels of slag present constructability problems in terms of early age strength and there have been instances of low scaling resistance offered by high volume slag concretes (Thomas and Innis 1998, Malvar et al. 2001).

With regards to literature related to the effectiveness of fly ash and slag in mitigating ASR by alkali-acetate and alkali-formate deicers, there seems to be no research conducted from this standpoint other than the work presented in this dissertation. However, there has been some research using SCMs like high reactivity metakaolin (HRM), silica fume and rice husk ash (RHA) to explore their mitigation potential in deicer exposure conditions.

Katkar (Katkar2006) used two reactive aggregates and two SCMs- high reactivity metakaolin (HRM) and silica fume, and conducted various mortar bar tests in the presence of potassium acetate, sodium acetate and sodium formate deicer. Results from this study indicated that the mitigation potential of HRM and silica fume was aggregate and deicer specific. But in general HRM proved to be more effective than silica fume when used at 12.5% cement replacement by controlling the expansions within the acceptable limit of 0.01% at 14 days in Modified ASTM C 1567 test. This study also proposed the use of a modified ASTM C 227 test for evaluating the efficacy of mineral admixtures in mitigating

ASR by simulating 'true' field conditions where concrete will be regularly exposed to deicing chemicals.

Wingard (Wingard 2007) used rice husk ash (RHA) to explore it as an ASR mitigation alternative for mortars and concretes exposed to 50% wt. commercial grade potassium acetate deicer. He used RHA at 5%, 10%, 15% and 20% cement replacement levels using one reactive aggregate and conducted various standard and modified ASTM C 1567 tests with as 1N NaOH and potassium acetate as soak solution, respectively. Results from his research concluded that rice husk ash was not only ineffective in reducing the ASR expansions; it led to increased expansions on increasing the RHA dosage. However, there was a reduction in the permeability of concrete samples, as observed in the results of the rapid chloride ion permeability test; and increase in the compressive strength with the same dosages of RHA. This study confirmed that the reaction mechanisms with potassium acetate deicer are different compared to 1N NaOH and hence, the test methods and theories which apply towards evaluating aggregates for their ASR potential and SCMs for their ASR mitigation potential, need not hold true with potassium acetate.

Limiting the alkali content of the cement and/or total alkali content of the concrete is another alternative to minimize the risk of ASR. However, this alternative has opinions divided among researchers based on their respective studies. It is recommended to limit the alkali content of cement to 0.6% Na2O equivalent or 3 $kg/m^3$ specified by ASTM C150 in order to minimize the risk of visual cracking. (Malvar et al. 2001, AASHTO 2000, CAS 2006). However, with regards to mortar and concrete samples made low alkali and high alkali cements and exposed to alkali-acetate and alkali-formate deicers, the alkali content of the cement did not have much influence on controlling the deleterious expansions due to ASR (Rangaraju et al.2007).

Lithium salts have been found to counter the aggregate reactivity and suppress the deleterious expansions and among the lithium compounds, lithium nitrate is the only compound that is believed not to exhibit a pessimum effect (Touma et al. 2001, Folliard et al. 2003, and Thomas et al. 2001). A research study was conducted by Rangaraju and Santhanam (2006) to explore the ASR mitigation potential of lithium nitrate when concrete is exposed to potassium acetate airfield deicer. In this study, the effectiveness of 30% aqueous solution of lithium nitrate in mitigating ASR was evaluated with four different types of reactive aggregates obtained from across the United States. In addition, the study explored the effectiveness of use of lithium compounds as a topical treatment for mitigating ASR in existing concrete. The findings of their study concluded that lithium nitrate was effective in suppressing the ASR expansions when a sufficient molar ratio of Li/Na is maintained in the cementitious matrix; which was found to be 0.74. This ratio was effective for three of the four reactive aggregates under study and was in conformity wit previous lithium mitigation studies. However, in some highly reactive aggregates, Li/NA ratio of 1.0 was required to suppress the expansions. The use of lithium nitrate as a topical treatment for ASR mitigation in existing concrete did not prove to be effective based on the experimental program conducted in this study.

## 2.7 Reaction Mechanisms of Alkali-Acetate and Alkali-Formate Deicers

Recently, Stark et al. (Stark et al. 2006) conducted a research study to develop an 'ASR performance test for concretes', known as the FIB (F.A. Finger-Institut für

Baustoffkunde) cyclic climatic storage test, and demonstrated that the alkali based deicers like the traditionally used NaCl and the new generation potassium acetate have the potential to accelerate ASR in concrete. The findings of this research confirm the findings of the research for FAA project 03-9 (Rangaraju et al.2007) that alkali-acetate and alkali-formate based deicers can cause deleterious ASR and also that their reaction mechanisms are different than the chloride based deicers. Based on the research study, Stark et al. has proposed two different possible mechanisms for the alkali-acetate and alkali-formate based deicers that lead to deleterious ASR. Mechanisms for formates and acetates are the same in principle, but due to the high solubility of formate salts, the reactions occur faster compared to acetates.

The first mechanism proposed is that of sudden increase in the pH of potassium acetate or potassium formate solution on contact with Portlandite ($Ca(OH)_2$). This was also observed in another study by Sompura (Sompura 2006). In both these studies (Stark 2006 and Sompura 2006) a sudden increase in the pH of the potassium acetate solution on addition of $Ca(OH)_2$ was observed and the pH kept on increasing before reaching a saturation level concentration.

$$K^+ + CH_3COO^- + H_2O \leftrightarrow CH_3COOH\ (aq.) + K^+ + OH^- \qquad \textit{(eq. 1)}$$

It is believed that this sudden increase is due to the formation of calcium acetate or calcium formate (in case of potassium formate solution) complex causes a drop in the concentration of the $Ca^{2+}$ ion which in turn leads to dissolution of new portlandite into the solution to maintain an equilibrium state. The dissolution of Portlandite releases new calcium

($Ca^{2+}$) and hydroxide ions (OH-) leading to an increase in the pH of the solution (eq. 2). However, as the pH increases the solubility of Portlandite decreases and the pH increase is possible only as long as there is an availability of free $Ca^{2+}$ ions in the solution and they can come in contact with the acetate or formate ions.

$$2K^{+} + 2CH_3COO^{-} + Ca^{2+} + 2\ OH^{-} \leftrightarrow\ 2Ca(CH_3COO)_2(aq.) + 2K^{+} + 2OH^{-} \quad \textit{(eq. 2)}$$

The second mechanism proposed by Stark et al. based on the microbiological degradation of the acetate or formate ions. This degradation increases with an increase in temperature, but increased degradation is possible even at lower temperatures (eq. 3).

$$2K^{+} + 2CH_3COO^{-} + 4O_2 \rightarrow 2K^{+} + CO_3^{2-} + 3H_2O + 3CO_2\ (\uparrow) \quad \textit{(eq. 3)}$$

When these degradation products come in contact with the pore solution or a saturated Portlandite solution, it forms a poorly soluble calcium carbonate precipitate (eq. 4). This results in a drop in the calcium ion concentration in the pore solution which in turn leads to the further dissolution of Portlandite to maintain equilibrium. The dissolution of Portlandite also adds up the OH- ion concentration and hence an increase in the pH.

$$2K^{+} + CO_3^{2-} + Ca^{2+} + 2\ OH^{-} \leftrightarrow 2K^{+} + 2\ OH^{-} + CaCO_3\ (\downarrow) \quad \textit{(eq. 4)}$$

Whatever mechanism occurs, it ultimately leads to (a) pH increase of the pore solution and (b) increase in the alkali concentration in the pore solution. Also, to exacerbate the situation, the solubility of potassium acetate (2530 g/l) and potassium formate (3310 g/l) in water is about nine times that of the commonly used pavement deicer NaCl (360 g/l), making the potassium acetate (or formate) highly hygroscopic and provides easy ingress of moisture and dissolved alkalis in concrete.

# CHAPTER III MATERIALS AND EXPERIMENTAL PROCEDURES

## 3.1 General

This chapter describes the experimental materials, test procedures and the test matrix involving these materials used in this study. This chapter also provides the reasons behind the selection of the materials and tests methods. The tests are broadly classified into three categories: Mortar tests, Concrete tests and other material characterization tests related to the aggregates, fly ashes, deicers and reagents.

## 3.2 Materials

The materials used in this study were commercial potassium acetate (KAc) deicer, reagent grade sodium hydroxide (NaOH), four reactive and two non-reactive aggregates from different sources, one type-I high alkali cement , fifteen fly ashes from different sources, selected based on their lime (%CaO) content, and one grade 120 slag. This study also involved using fused silica and hydrated lime for specific studies related to silica dissolution.

### 3.2.1 Deicers and Reagents

A commercial grade potassium acetate deicer having a concentration of 50% wt. solution (6.4 molar) was used as a soak solution in all the modified ASTM C 1260, C 1567 and C 1293 tests. The deicer was supplied by Cryotech Deicing Technology under the brand name 'E36® Liquid runway Deicer'. The properties of the deicer used, as reported by the manufacturer, are presented in Table 3.1.

Table 3.1 Properties of Cryotech E36® Liquid Runway Deicer

| Property | Description |
|---|---|
| Composition | 50% aqueous potassium acetate solution, by weight |
| Appearance | Clear, colorless (blue if indicator dye is used) |
| Density | 1.282 g.cm3 (at $20^{0}$C/$68^{0}$ F) |
| Viscosity | 10 cp maximum (at $20^{0}$C/$68^{0}$F)<br>20 cp maximum (at $0^{0}$C/$32^{0}$F) |
| Flash point | Non-flammable |
| Freezing Point | $-60^{0}$C/ $-70^{6}$F |
| pH | 11.0 ±0.5 (at $25^{0}$C) |
| Specific Gravity | 1.25 to 1.30 (at $20^{0}$C) |

*Note: Information as provided by the manufacturer*

Among all the alkali-acetate and alkali-formate deicers potassium acetate deicer was selected due to the fact that it is the most widely used airfield pavement deicer across the United States (Rangaraju et al. 2006). The selection was also based on the findings of a previous study conducted at Clemson University which concluded that potassium acetate has a significant potential in causing ASR (Rangaraju and Olek 2007).

In real life this deicer is used at the same concentration as used in this study. In routine deicing applications on bare pavement surface, the melting of the snow and ice may cause the dilution of the applied deicer. However, the freezing-thawing and wetting-drying cycles that the concrete pavement undergoes, along with the repeated deicing applications, presents a situation where the pore solution of the concrete could be saturated with the potassium acetate salt.

The other salt used in this study is sodium hydroxide (NaOH). A reagent grade sodium hydroxide in pellet form was used to prepare a 1 normal (1N) solution. This 1N

NaOH solution was used as a soak solution for all the standard ASTM C 1260 and ASTM C 1567 tests and for Modified ASTM C 1293 tests.

### 3.2.2 Aggregates

Six aggregates were used in this study and were selected to represent various levels of alkali-silica reactivity. Four of the six aggregates are characterized as reactive aggregate, and the remaining two aggregates are non-reactive in nature. The details of the four reactive aggregates are as follows:

1) New Mexico Rhyolite- This aggregate is one of the most reactive aggregates found and it primarily consists of Rhyolite as the reactive component. This aggregate is a gravel from Las Placitas Gravel pit in Bernalillo county in New Mexico. (Touma et al. 2001, Barringer 2000).
2) Spratt Limestone – This aggregate has an established history of being alkali-silica reactive and has been used as a reference aggregate in numerous ASR research studies (Rangaraju et al. 2006). The source of this aggregate is from Ontario, Canada and is quarried from the Spratt quarry. It primarily consists of calcite with minor amounts of dolomite and about 10% insoluble residue. The alkali-silica reactive component of the rock is reported to be 3%-4% microscopic chalcedony and black chert, which is finely dispersed in the matrix (Rogers 1999).
3) North Carolina Argillite- This aggregate primarily consists of reactive argillite/metatuff and its source is from Goldhill Quarry in North Carolina. This aggregate has an established field history of being alkali-silica reactive in several bridge structures across North Carolina (Leming et al. 1996)

4) South Dakota Quartzite- This aggregate primarily consists of strained quartz grains cemented with interstitial secondary quartz cement. The interstitial matrix also consists of microcrystalline quartz, hematite and kaolinite. This aggregate is quarried from L.G.Everist Quarry in Dell Rapids, South Dakota. This is a moderate-to-slow reactive aggregate and has an established history of being alkali-silica reactive in concrete pavements in Minnesota and South Dakota (Rangaraju 2000, Johnston et al. 2000)

The details of the two non-reactive aggregates are as follows:

1) Illinois Dolomite- This aggregate has an established field history of being non-reactive and it primarily consists of dolomite. It is quarried stone from Thornton quarry in Illinois.
2) Ottawa Sand- This is non-reactive silica sand conforming to ASTM C 778 and is 99.7% silicon dioxide. This sand is produced by the US Silica Company and its principal mineral is quartz. This sand is used as fine aggregate in the modified ASTM C 1293 tests.

The physical properties of all the six aggregates are presented in Table 3.2.

Table 3.2 Properties of Aggregates

| Aggregate Property | Spratt, Limestone | SD, Quartzite | NC, Argillite | NM, Rhyolite | IL, Dolomite | IL, Ottawa |
|---|---|---|---|---|---|---|
| Water absorption,% | 0.456 | 0.42 | 0.344 | 1.087 | 2.12 | 0.0 |
| Bulk specific gravity | 2.69 | 2.51 | 2.75 | 2.60 | 2.66 | 2.65 |
| Bulk specific gravity (SSD) | 2.706 | 2.52 | 2.76 | 2.63 | 2.71 | 2.65 |
| Dry rodded Unit weight, $kg/m^3$ | 1568.3 | 1557.62 | 1566 | 1585.25 | 1563.7 | --- |

### 3.2.3 Cement

A high alkali cement (Type I) with a $Na_2O$ equivalent of 0.82% ($Na_2O_{eq}$) and an autoclave expansion of 0.08% was used for this study. The source of the cement was from Lehigh plant in Evansville, PA. The chemical composition of this cement is provided in Table 3.3. This cement was used for all the concrete and mortar tests in this study.

Table 3.3 Chemical Composition of Type I High Alkali Cement

| **Oxide, %** | | | |
|---|---|---|---|
| $SiO_2$ | 19.74 | $Na_2Oeq = Na_2O + 0.68K_2O$ | 0.82 |
| $Al_2O_3$ | 4.98 | $K_2O$ | -- |
| $Fe_2O_3$ | 3.13 | Loss on Ignition (LOI) | 1.9 |
| CaO | 61.84 | Insoluble Residue | 0.25 |
| MgO | 2.54 | C3A | 8 |
| $SO_3$ | 4.15 | C3S | 52 |
| Available Alkali | -- | | |

### 3.2.4 Fly Ashes

Fifteen fly ashes that differed in their lime contents were selected for evaluation in this study. Based on ASTM C 618 specification, fly ashes are classified into two categories-Class 'C' and Class 'F'- based on their content of SiO2, Al2O3 and Fe2O3. The Canadian standards (CSA A3001, 2003) provide an alternate fly ash classification based on their lime (% CaO) content. The three categories are as follows: High Lime (CH) fly ash (CaO >20%), Intermediate Lime (CI) fly ash (Ca0 8-20%) and Low Lime (F) fly ash (CaO <8%). Since in this study the selection of the fly ashes was based on the lime content, the Canadian standard for fly ash classification seemed more appropriate and as per that classification, the study involved using 4 high lime (HL), 6 intermediate lime (IL) and 5 low lime (LL) ashes. Of the 15 fly ashes, 10 were from different plants of Boral Material Technologies, 4 from Headwater Resources and one from Southeastern Fly Ash Company (SEFA). The chemical compositions of the three classes of fly ashes are provided in table 3.2, table 3.3 and table 3.4. All the 15 fly ashes were tested in the mortar bar test at 25% cement replacement by mass. Three fly ashes (HL3, IL5 and LL3), one representing each of the three classes, were tested at 15% and 35% cement replacement levels, in addition to the 25% replacement, with all the four reactive aggregates and one non-reactive aggregate to understand the influence of fly ash dosage on ASR mitigation potential. All selected fly ashes conform to the ASTM C618 specifications.

Table 3.4 Chemical composition of High Lime fly Ashes

| | Fly Ashes (High Lime) | | | |
|---|---|---|---|---|
| Oxide (%) | HL1 | HL2 | HL3 | HL4 |
| Silicon Dioxide ($SiO_2$) | 39.66 | 32.44 | 34.55 | 31.31 |
| Aluminum Oxide ($Al_2O_3$) | 20.42 | 19.31 | 18.10 | 18.64 |
| Iron Oxide ($Fe_2O_3$) | 5.51 | 8.19 | 5.68 | 5.49 |
| Sum of $SiO_2$, $Al_2O_3$, $Fe_2O_3$ | 65.59 | 59.94 | 58.33 | 55.44 |
| Calcium Oxide (CaO) | 22.85 | 27.47 | 27.5 | 29.85 |
| Magnesium Oxide (MgO) | 4.22 | 5.19 | 5.04 | 5.54 |
| Sulfur Trioxide ($SO_3$) | 1.21 | 2.12 | 2.80 | 2.55 |
| Sodium Oxide ($Na_2O$) | 1.49 | 1.11 | 1.59 | 1.88 |
| Potassium Oxide ($K_2O$) | 0.69 | 0.42 | 0.36 | 0.32 |
| Total Alkalies (as $Na_2O$) | 1.90 | 1.39 | 1.83 | 2.09 |
| Available Alkalies (as $Na_2O$) | 0.95 | 0.83 | NA | 1.34 |
| Loss on Ignition, % | 0.27 | 0.06 | 0.18 | 0.23 |
| Specific Gravity, g/cc | - | 2.73 | 2.63 | 2.77 |

Table 3.5 Chemical composition of Intermediate Lime Fly Ashes

| | Fly Ashes (Intermediate Lime) | | | | | |
|---|---|---|---|---|---|---|
| Oxide (%) | IL1 | IL2 | IL3 | IL4 | IL5 | IL6 |
| Silicon Dioxide ($SiO_2$) | 52.40 | 52.97 | 52.92 | 56.26 | 49.69 | 41.91 |
| Aluminum Oxide ($Al_2O_3$) | 23.20 | 22.25 | 21.3 | 19.88 | 15.03 | 21.08 |
| Iron Oxide ($Fe_2O_3$) | 5.73 | 5.39 | 7.51 | 4.48 | 6.6 | 5.61 |
| Sum of $SiO_2$, $Al_2O_3$, $Fe_2O_3$ | 81.33 | 80.61 | 81.73 | 80.62 | 71.32 | 68.60 |
| Calcium Oxide (CaO) | 10.33 | 10.45 | 10.56 | 12.25 | 15.63 | 18.94 |
| Magnesium Oxide (MgO) | 2.08 | 2.33 | 2.83 | 2.76 | 4.92 | 4.21 |
| Sulfur Trioxide ($SO_3$) | 0.64 | 0.52 | 0.49 | 0.48 | 0.90 | 0.98 |
| Sodium Oxide ($Na_2O$) | 1.26 | 0.94 | 0.57 | 0.66 | 2.53 | 2.15 |
| Potassium Oxide ($K_2O$) | 0.97 | 1.1 | 1.38 | 0.83 | 2.13 | 0.67 |
| Total Alkalis (as $Na_2O$) | 1.9 | 1.66 | 1.48 | 1.21 | 3.93 | 2.59 |
| Available Alkalis (as $Na_2O$) | 0.69 | 0.54 | NA | 0.34 | NA | 1.17 |
| Loss on Ignition, % | 0.75 | 0.78 | 0.11 | 0.29 | 0.01 | 0.54 |
| Specific Gravity, g/cc | 2.26 | 2.4 | - | 2.41 | 2.55 | 2.57 |

Table 3.6 Chemical composition of Low Lime Fly Ashes

| | Fly Ashes (Low Lime) | | | | |
|---|---|---|---|---|---|
| Oxide (%) | LL1 | LL2 | LL3 | LL4 | LL5 |
| Silicon Dioxide ($SiO_2$) | 52.78 | 54.12 | 58.67 | 54.53 | 52.4 |
| Aluminum Oxide ($Al_2O_3$) | 27.46 | 27.79 | 20.86 | 26.29 | 23.2 |
| Iron Oxide ($Fe_2O_3$) | 8.90 | 8.01 | 11.51 | 5.03 | 5.73 |
| Sum of $SiO_2$, $Al_2O_3$, $Fe_2O_3$ | 89.14 | 89.92 | 91.04 | 85.85 | 81.33 |
| Calcium Oxide (CaO) | 1.27 | 1.34 | 3.35 | 7.31 | 7.49 |
| Magnesium Oxide (MgO) | 1.08 | 0.90 | 1.15 | 1.60 | 1.71 |
| Sulfur Trioxide ($SO_3$) | 0.12 | 0.16 | 0.40 | 0.39 | 0.80 |
| Sodium Oxide ($Na_2O$) | NA | 0.29 | 0.46 | 0.27 | 0.41 |
| Potassium Oxide ($K_2O$) | NA | 2.79 | 1.12 | 1.05 | 1.16 |
| Total Alkalis (as $Na_2O$) | NA | 2.13 | 1.20 | 0.96 | 1.17 |
| Available Alkalis (as $Na_2O$) | 0.67 | 0.56 | NA | 0.25 | 0.2 |
| Loss on Ignition, % | 2.97 | 2.51 | 0.036 | 0.73 | 0.73 |
| Specific Gravity, g/cc | 2.27 | 2.26 | 2.44 | 2.17 | 2.49 |

### 3.2.5 Ground Granulated Blast Furnace Slag

A Grade 120 slag was used in this study at 40% and 50% cement replacement level by mass. The chemical composition of slag is provided in table 3.5. and it conforms to the chemical requirements of ASTM C 989.

Table 3.7 Chemical Composition of Slag

| **Oxide, %** | | | |
|---|---|---|---|
| $SiO_2$ | 38.17 | Available Alkali | --- |
| $Al_2O_3$ | 7.31 | Loss on Ignition (LOI) | --- |
| $Fe_2O_3$ | 0.78 | $Na_2O_{equivalent}$ | --- |
| CaO | 39.12 | $K_2O$ | 0.34 |
| MgO | 12.48 | Insoluble Residue | --- |
| $SO_3$ | 2.56 | $TiO_2$ | 0.78 |
| $Mn_2O_3$ | 0.40 | Specific Gravity, g/cc | 2.92 |

### 3.3 Experimental Methods

The experimental methods are broadly classified into three categories: mortar tests, concrete tests and other tests related to characterization of aggregates, fly ashes, deicers and reagents. The mortar bar tests include the standard and modified versions of ASTM C 1260 and ASTM C 1567 tests, while the modified ASTM C 1293 tests comprise of the concrete prism tests. All the mortar and concrete samples were tested for their dynamic modulus of elasticity using the impulse excitation technique. To study the microstructure of the samples, scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis were conducted.

#### 3.3.1 Standard ASTM C 1260 Test Procedure

The standard ASTM C 1260 test known as "Accelerated Mortar Bar Test (AMBT)" is a method to assess the reactivity of aggregates. In this test, mortar bars (25 mm x 25 mm x 285 mm) with gage studs at ends are prepared at a water-to-cement ratio of 0.47. The aggregate-to-cement ratio, by mass, is maintained at 2.25. After 24 hours of curing in a moist room, the mortar bars are demolded and transferred into a storage container with sufficient water to immerse all samples. The sealed container is placed in an oven at 80ºC for 24 hours. After 24 hours, the mortar bars are removed from the oven and a zero reading (0 day reading) is taken. The mortar bars are subsequently transferred into a 1 N sodium hydroxide solution, which is preheated to 80ºC. Length change readings are taken thereafter at periodic intervals to determine the percent expansion. According to the specifications, this test runs for 14 days (excluding 2 days of initial conditioning). However, in this research, the length-change measurements were taken up to either 28 days or 56 days at intervals of 0, 3, 7, 11, 14, 21, 28, 42 and 56 days in order to study the effect of prolonged exposure to deicer solutions on the expansions.

Four mortar bars per test were used in each test. A ratio of the soak solution to the mortar bars was 4.5:1 (by volume) was maintained for all the standard and modified mortar bar tests. As per ASTM C 33, a mortar bar expansion of 0.1% or less at 16 days in the standard ASTM C 1260 test (i.e. 14 days of exposure to soak solution) is considered to indicate the innocuous nature of the aggregate, in other words 'non-reactive'. Mortar bar expansion greater than 0.2% at 16 days is considered to indicate the potentially reactive nature of the aggregate. Mortar bar expansion between 0.1% and 0.2% is considered to be inconclusive about the ASR reactivity of the aggregate. When adequate field performance data is not available to ascertain the reactivity of such aggregates, it is recommended that they require an additional evaluation using the concrete prism test (ASTM C 1293).

The acceptance limits for the expansion of mortar bars and their classification as being reactive or non-reactive vary from region to region. FAA Advisory Circular 150/5370-10B – Standard for Specifying Construction of Airports – considers aggregate to be reactive, when expansions in mortar bars subjected to standard ASTM C 1260 test exceed 0.1% at 16 days (FAA 2005a). However, the revised AC 150/5370-10B specification employed by the Northwest Mountain Region Airports Division considers aggregate to be reactive if the expansion of mortar bars in the standard ASTM C 1260 test exceeds 0.1% at 30 days after casting (i.e. 28 days of exposure to soak solution)- (FAA (2005b). Airports anticipating the use of deicers in this region implement a tighter specification limit and the aggregate is considered reactive if expansion of mortar bars in the modified ASTM C 1260 (deicer used as soak solution instead of 1 N NaOH) test exceeds 0.08% at 30 days after casting. The tighter specifications may be justified on airfield pavements, where concerns and consequences arising from Foreign Object Debris (FOD) are serious.

In this study the ASTM C 33 aggregate acceptance limit of 0.1% mortar bar expansion at 14 days was used to classify aggregates as 'innocuous' for both standard and modified tests. However, the expansion results will also be evaluated against the more stringent expansion limits imposed by the revised AC 150/5370-10B standards specified by the Northwest Mountain Regions Airport Division, where applicable.

### 3.3.2 Modified ASTM C 1260 Test Procedure

The modified ASTM C 1260 test uses the same procedures and materials for casting, demolding, storing and intervals for taking the length change readings. However, the only difference between the standard and modified test is the soak solution used in the test. The standard test uses 1 N NaOH as soak solution, whereas the modified test uses a 6.4 M solution of potassium acetate deicer. The volume of the soak solution is the same in both the versions of the ASTM C 1260 test.

### 3.3.3 Standard ASTM C 1567 Test Procedure

This test is used to evaluate the potential of ASR for combinations of cementitious materials and aggregate. All the aspects of testing for this test are same as the standard ASTM C 1260 test except for the replacement (by mass) of a portion of Portland cement by supplementary cementitious materials like fly ash and slag. In this study fly ash was used at three replacement levels, 15%, 25% and 35% by mass of cement, and slag at 40% and 50% levels. The soak solution for this test is a 1 N NaOH solution.

### 3.3.4 Modified ASTM C 1567 Test Procedure

The modified version of the ASTM C 1567 test involves using the same test materials and regimen as for the standard test. However, the only difference between the standard and modified tests was the soak solution for the mortar bars. The standard test uses 1 N NaOH as soak solution, whereas the modified test uses a 6.4M solution of potassium acetate deicer.

### 3.3.5 Modified (Type-1) ASTM C 1293 Test Procedure

The ASTM C 1293, also known as the "Concrete Prism Test" is the standard test method for determination of length change of concrete due to alkali silica reaction. To explain the modified versions of the standard test, the standard test is briefly described. The standard test involves preparation of concrete prisms (75mm x 75mm x 285mm) with gage studs at the ends. The concrete is made using high alkali cement, the aggregate in question and a supplementary non-reactive coarse or fine aggregate. The alkali content of concrete is raised to achieve 1.25% $Na_2O$eq. by adding NaOH to the mix water. After initial curing for 24 hours, the concrete prisms are placed in 5 gallon pails on specially fabricated stands to hold three prisms vertically and above the pail bottom surface. The pail is filled with water to about half inch level from the bottom, but not touching the prisms placed in the stand, to create a humid environment within the bucket. The pail is sealed and stored in a 38$^{o}$C temperature controlled storage room and length change readings are taken at periodic intervals up to 12 months (24 months in case of concrete with SCMs).

In the modified (Type-1) ASTM C 1293 test the concrete prisms are stored in a shoe box type container, typically used in the mortar bar tests, and are soaked in 1N NaOH solution instead of storing them in 100% relative humidity environment. Also, the prisms are stored in a horizontal direction unlike the vertical placement in the standard test. This modified test procedure has been investigated as a part of the FAA 03-9 project conducted at Clemson University. For this research study, the concrete mixes were made using SCMs at different cement replacement levels- 3 different fly ashes at 25%, 35% cement replacement levels and slag at 40% and 50% cement replacement. Both the SCMs were tested with two reactive aggregates and one non-reactive aggregate. The alkali content of the concrete was boosted to 1.25% of the mass of cement by adding NaOH to the mix water, while the alkalis from the fly ashes were not considered towards this calculation of 1.25% $Na_2Oeq$. The fine aggregate used in the concrete was a non reactive graded Ottawa sand confirming to ASTM C 778. Four concrete prisms were made for each mix and one of the four prisms was used as a sacrificial specimen to study the changes in the microstructure at different intervals during the test regimen. Length change readings were taken at every month up to 12 months followed by once every 3 months up to another 12 months to determine the percent linear expansion.

The expansion limits for this test are set at the 2 year (1 year in case of concrete without SCMs) expansion measurement. The aggregate is considered as non-reactive if the percent expansion at 2 years is less than or equal to 0.04% whereas it is considered to have a strong negative interaction between the concrete and the soak solution (1N NaOH or deicer) if it is more than 0.04%.

### 3.3.6 Modified (Type-2) ASTM C 1293 Test Procedure

The only difference between the Type-1 and Type-2 modification of the ASTM C 1293 prism is the soak solution used in immersing the concrete prisms. Type 1 version of the test used 1N NaOH as soak solution whereas Type 2 version uses a 6.4 molar concentration potassium acetate deicer solution as soak solution. The remaining test parameters are the same for both the modified versions of the test.

### 3.3.7 Dynamic Modulus of Elasticity- Impulse Excitation Technique

To quantify the physical distress occurring in the mortar and concrete specimens during their test regimens, dynamic modulus of elasticity (DME) measurements were taken at periodic intervals. The DME gives an idea of the integrity of the internal structure of the specimens subjected to the 1N NaOH and potassium acetate deicer solutions. The DME values were determined using the resonant frequency method based on impulse excitation technique based on ASTM E 1876-01 test procedure. A GrindoSonic™ instrument was used to determine the resonant frequencies of the mortar bars and concrete prisms.

In this test, the mass and the resonant frequency of the mortar and concrete specimens were determined soon after taking the length-change measurements. The dimensions of the mortar bars and concrete prisms were assumed to be constant and the effects of the metal gage studs at the ends of the bars and prisms were neglected, as it was a common factor for all measurements. DME values were calculated for the same ages at which length-change measurements were made. Changes in DME values were correlated with expansion measurements to understand the progressive deterioration in stiffness of the mortar and concrete specimens.

### 3.3.8 Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Analyses

SEM in back-scattered mode and EDX analyses was conducted on polished sections of mortar bars using either an ASPEX SEM Instrument or Hitachi 3400N. The instrument was operated at an accelerating voltage of 20KeV.

The samples from selected mixes of the ASTM C 1567 (standard and modified) and ASTM C 1293 (modified type-2)for the SEM-EDX were obtained by slicing the mortar bars and concrete prisms using a masonry saw followed by a slow-speed diamond saw. These samples were cleaned in propanol using an ultrasonic cleaning system so as to get rid of the residue formed due to the slicing process. This was followed by drying them at 38° C, cooling to room temperature and then embedding them using a Low Viscosity Epoxy. The epoxy embedded samples were then polished on a series of diamond embedded discs with progressively increasing the fineness in the following order: #60, #140, #600 and #1200 grit (mean particle size of 5 microns). The final polish of the samples was done using diamond suspensions of 3 micron, 1 micron and 0.25 micron on a polishing cloth pad at 150 RPM.

### 3.3.9 pH Measurements

The pH of the soak solutions before and after the test regime was measured to understand the mechanisms between the mortar and concrete specimens and the soak solutions (NaOH and potassium acetate). As described in chapter 2, it is recognized that the occurrence of ASR is facilitated by a high pH environment, meaning a high concentration of $OH^-$ ions in the solutions. Hence, to study the influence of the mortar and concrete specimens made using SCMs with the deicer and NaOH solution, it becomes necessary to monitor the pH and see if a correlation exists between the pH and expansions observed during the tests.

pH experiments were also conducted on the interaction of cement-fly ash, cement-slag and control cement paste samples with 1N NaOH and potassium acetate solution. The cement-SCM and control paste samples were made at the same w/c ratio used in the ASTM C 1260 and C 1567 test, i.e. 0.47. The paste samples were cast in cylindrical polypropylene plastic vials and allowed to set for 1 day following which they were demolded from the vials and soaked in 1N NaOH and potassium acetate solutions in polypropylene plastic bottles. Two vial shaped cement paste samples were cast for each combination of cement-fly ash, cement-slag and control mixes. Three fly ashes (Low lime, Intermediate Lime and High lime) were tested at 15%, 25% and 35% dosages and a slag was tested at 40% dosage. pH measurements were taken at 0, 3, 7, 14 and 21 days after soaking the samples in the solutions.

The pH of the soak solution was determined using an Oakton pH 110 meter with a low-sodium error and a high salt glass electrode, calibrated to buffer solutions with pH 7.0, 10.05, and 12.45.

### 3.3.10 Silica Dissolution Study- Inductively Coupled Plasma (ICP) Test

Despite the enormous research effort spent on ASR over the last three decades, the issue of undesired silica dissolution of the aggregate is still not fully understood. To understand the influence of potassium acetate on the silica dissolution process, a series of short term tests were conducted. This experiment was conducted following the analysis of results of some of the concrete prism and mortar bar samples soaked in potassium acetate

(Modified ASTM C 1567 and Modified ASTM C1293) where it was found that though there were high expansions and severe cracking of the samples, when these samples were observed under a scanning electron microscope the aggregate particles did not show any major signs of cracking. This led to a hypothesis that some amount of reactive silica from the reactive aggregate particles might be disintegrating and dissolving in the presence of pore solution and potassium acetate and lead to deleterious ASR. This study was hence conducted to prove/disprove this hypothesis.

Since the objective was to understand the influence of potassium acetate on silica dissolution in a high pH environment, the use of natural aggregates was avoided as it would complicate the situation by inducing variability due to the type of aggregate selected. Instead, a crushed and sieved (passing ASTM#50 (300μ) sieve and retained on ASTM#100(150μ) sieve) fused silica was used to represent a reactive siliceous aggregate.

Two parallel tests were conducted for both 1N NaOH solution and potassium acetate deicer to study the influence of potassium acetate deicer on dissolution of silica over fixed time intervals. One test had fused silica and potassium acetate in the proportions 10 gram: 50 ml. The other test had the same proportions of fused silica and potassium, but 1gram $Ca(OH)_2$ was added to the solution. $Ca(OH)_2$ represented the portlandite produced during the hydration of the cement paste and this leads to a high pH in the pore solution. It was hypothesized that the Portlandite produced on hydration of the cement paste of the mortar bars, will react with the potassium acetate leading to a rise in the pH. Past research (Sompura 2006) using the same potassium acetate deicer and lime had concluded that the addition of 0.1 to 0.7 grams of $Ca(OH)_2$ to 50 ml potassium acetate (6.4M) led to a sudden increase in the pH of the solution (from 11.04 to ~14.21 in 15seconds). This study was used as our basis for determining the proportion of deicer and $Ca(OH)_2$. Samples were prepared and stored in small plastic bottles for a fixed period of time after which the solution s were filtered using a filter paper and the filtered solution was analyzed for four elements (Si, K, Na and Ca) by ICP technique. Separate samples were prepared for each age for 8.5, 26, 48, 168, 384 and 504 hours.

A duplicate set of these tests were conducted by storing the samples in a hot room at 38°C controlled temperature environment. The objective of this test was to study the influence of temperature on silica dissolution. The test matrix for the ICP tests is presented in table 3.8.

Table 3.8 Test Matrix for ICP Tests

| Hours Stored | 1N NaOH (Room Temp) | | 1N NaOH (38°C) | | Pot. Acetate (Room Temp.) | | Pot. Acetate (38°C) | |
|---|---|---|---|---|---|---|---|---|
| | Si | Si + CH | Si | Si + CH | Si | Si + CH | Si | Si + CH |
| 8.5 | X | X | X | X | X | X | X | X |
| 26 | X | X | X | X | X | X | X | X |
| 48 | X | X | X | X | X | X | X | X |
| 168 | X | X | X | X | X | X | X | X |
| 384 | X | X | X | X | X | X | X | X |
| 504 | X | X | X | X | X | X | X | X |
| 672 | X | X | X | X | X | X | X | X |

*Note: Si: Fused Silica, CH:* $Ca(OH)_2$

A JY ULTIMA 2 (Horiba Jobin Yvon, Longjumeau, France) sequential ICP spectrometer was used to determine the elemental compositions of the filtered solutions of sodium hydroxide and potassium acetate. The elements analyzed by this technique were Si, Na, K and Ca. The operating parameters for the ICP tests are presented in table 3.9.

Table 3.9 Instrumental Parameters for ICP Tests

| Parameters | JY ULTIMA 2 ICP Spectrometer |
|---|---|
| Power (W) | 1200 |
| Gas (Argon):<br>Nebulizer<br>Coolant<br>Auxiliary | <br>0.02 L/min<br>16 L/min<br>0.2 L/min |
| Pump Speed | 20 rpm |
| Wavelengths (nm):<br>Si<br>Na<br>K<br>Ca | <br>252.412<br>588.995<br>766.490<br>211.276 |

Before each session of ICP elemental analysis, a calibration was done using external standards. Standard solutions were prepared by diluting 1000 mg/l multi-element standard to obtain concentrations of 0ppm, 2.5ppm, 5ppm, 7.5ppm and 10ppm. Calibration standards were run in 5 replicates and the values were plotted as a linear function of each element and the objective was to achieve more than 98%. Regression coefficient for each element calibrated. Since, the filtered solutions of NaOH and potassium acetate were highly concentrated, they were diluted to 50 times its original concentrations.

### 3.3.11 X-Ray Diffraction (XRD) Test

Fly ashes are heterogeneous fine powders consisting of mostly rounded spherical glassy particles and have varied amounts of silica, alumina and iron oxide content (Helmuth 1987).

The structure of fly ash in terms of the arrange of atoms and formation of crystalline and non-crystalline phases is governed by various factors such as, type of coal, combustion process used and the use of conditioning agents to aid extraction of fly ash by electrostatic precipitators (ESPs). Based on the mineralogical composition of the fly ashes and the conditions during cooling of fly ash particles when in a molten state, the phases are determined. It is important to know the phases of the fly ash to characterize and understand the influence of these phases on the chemical and physical properties of fly ashes. Fly ash consists of crystalline and non-crystalline (glassy) phases with a majority of the glassy phase (>70%) (Helmuth 1987). Class F (low lime fly ashes or alumina silica fly ashes) and Class C fly ashes have crystalline phases characterize of their type, with the high lime fly ash having lower glass content. Class C fly ashes have appreciable quantities of free CaO with some CaO encapsulated in the glass.

X-Ray Diffraction technique has been used by many researchers to identify the crystalline and other phases in fly ash (Helmuth 1987). The XRD diffractograms provide useful information that distinguishes the low, intermediate and high lime fly ashes based on the phases identified. For this research, XRD was used as a qualitative tool to understand the behavior of fly ashes in ASR mitigation. XRD analyses were conducted on six of the fifteen fly ashes representative of the three fly ashes classes LL1 and LL3 (Low lime fly ashes), IL5 and IL6 (Intermediate lime fly ashes) and, HL3 and HL4 (High lime fly ashes). The chemical composition of these selected fly ashes is provided in tables 3.4 to 3.6.

Fly ashes were ground for approximately 5 minutes with an agate mortar and pestle and the ground ashes were mounted on an aluminum well shaped holder and the surface leveled. A Scintag 2000 system with a germanium detector and a seven-position automatic sample changer was used for powder diffraction. The X-Ray 2theta angle range was from $5^o$ to $70^o$ (Cu K$\alpha$ radiation) with a scan rate of $0.01^0$ per minute. The analysis of the peak intensities obtained from the XRD, to identify the best possible match for a crystalline phase, was done using the Inorganic Crystal Structure Database, NIST Crystal Data File and Powder Diffraction File electronic data base.

### 3.3.12 Characterization of Fly Ashes Using X-Ray Diffraction

This section presents the results of the XRD analysis performed on six fly ashes described in the previous section- 3.3.11.The X-Ray Diffraction results are presented in the form of diffractograms that show peaks of the various crystalline phases present in the fly ashes. The fly ashes are characterized based on these crystalline phases identified in the diffractograms.

Analyzing the patterns of the diffractograms and the peaks found in them, it is extremely difficult to identify all mineral phases present in the fly ash. This is due to the extensive overlapping of the peaks of the components making it difficult to conclude with certainty that a particular component is present.

In the diffractograms the following acronyms are used to represent the crystalline components: Quartz (Qz)-$SiO_2$, Melilite (Ml)- $2CaO.Al_2O_3.SiO_2$, $C_3A$- $3CaO.Al_2O_3$, Periclase

(Pc)-MgO, Klein's Compound (Kl)- $4CaO.3Al_2O_3.SiO_2$, Anhydrite (Ah)- $CaSO_4$, Lime (CaO), Gehlenite (Gh)- $Ca_2Al_2SiO_7$, Magnetite (Fe)-$Fe_3O_4$, Mullite (Mu)-$3Al_2O_3.2SiO_2$, Kalsilite (Ka)-$KAlSiO_4$.

## Low Lime Fly Ash

Figure 3.1 shows the XRD analysis of low lime fly ashes LL1 and LL2. The difference between the two XRD patterns is the intensity of the peaks observed. At 20.8 degree and 26.5 degree (2θ angle) the intensity of the quartz peak is higher in LL2 compared to LL1. However, the major peaks detected in both the fly ashes were same and were found at the same 2θ angle. The major crystalline phase observed in both these fly ashes were quartz(Qz), mullite (Mu), gehlenite (Gh), magnetite (M) and kalsilite (Ka). The presence of these phases confirms the information published in literature related to the crystalline phases typically found in low lime fly ashes.

## Intermediate Lime Fly Ash

Figure 3.2 shows the XRD analysis of intermediate lime fly ashes IL5 and IL6. The difference between the two fly ashes was the intensity of the quartz peak at around $26^0$ 2θ angle. Also, the presence of $C_3A$- $3CaO.Al_2O_3$ was seen only in IL6 that has a higher %CaO content of the two. Periclase (Pc) was not seen in IL5 but was detected in IL6. CaO peaks were seen in both the diffractograms which were absent in the two low lime fly ashes. Quartz (Qz), mullite (Mu) and anhydrite (Ah) - $CaSO_4$ peaks were also seen in both the fly ashes.

## High Lime Fly Ash

The major crystalline phase's characteristic of high lime fly ashes were all detected in HL3 and HL4 shown in figure 3.3. These include Anhydrite (Ah)- $CaSO_4$ , melilite (Ml), Periclase (Pc), $C_3A$, CaO, gehlenite (Gh) and kalsilite (Ka). However, Klein's compound (Kl) was observed only in HL3. Quartz (Qz) was detected in both the fly ashes just like all the other class fly ashes but HL3 had a higher intensity of (Qz) peak around $26^0$ 2θ angle.

In summary, the difference between the six fly ashes (three classes) was number of peaks, the intensity of the peaks and the crystalline phases detected in them. Low lime and high lime fly ashes had around 28 peaks detected while the intermediate lime fly ashes had only 19 detected peaks. The highest peak observed was the quartz (Qz) peak observed in LL2 followed by HL3. Phases like gehlenite (Gh) and kalsilite (Ka) are typically found in high lime fly ashes but these were detected in low lime (LL1 and LL2) and intermediate lime (IL5) fly ashes too. Similarly, mullite (Mu) and $C_3A$- $3CaO.Al_2O_3$ typically found in low and high lime fly ashes respectively, were detected in one intermediate lime fly ash (IL6) too. However, quartz was the only crystalline phase common to all the six fly ashes examined. It was interesting to note that intermediate lime fly ashes (IL5 and IL6) had some crystalline phases common to low and high lime fly ashes respectively.

Another significant difference between the low and high lime fly ashes is between the "diffraction hump" produced by the X-ray scattering from the glass structure. This broad "hump" can provide an indication of the character of the glass in the fly ash and it is useful to know this because most of the reaction of fly ash in concrete is usually attributable to the reaction of glass (Diamond 1981). This difference in the broad hump can be seen in figure 3.4 where the XRD patterns of all the six fly ashes are stacked up. It can be seen that the difference in the hump (region between $5^0$ 2θ and $33^0$ 2θ angle), though subtle, is noticeable. For low lime fly ashes the maximum intensity was found around $24^0$ 2θ angle (LL1-$24.2^0$, LL2-$24.1^0$) which is characteristic of siliceous fly ashes. Intermediate lime fly ashes had a maximum intensity around $24^0$ but it increased to $24.6^0$ as the %CaO content increased (IL5-$24.0^0$, IL6-$24.6^0$). High lime fly ashes have a flat hump with a skewed pattern that peaks around $32^0$ 2θ (HL3-$32.22^0$, HL4-$32.4^0$).This skewed pattern is characteristic of the high lime fly ashes.

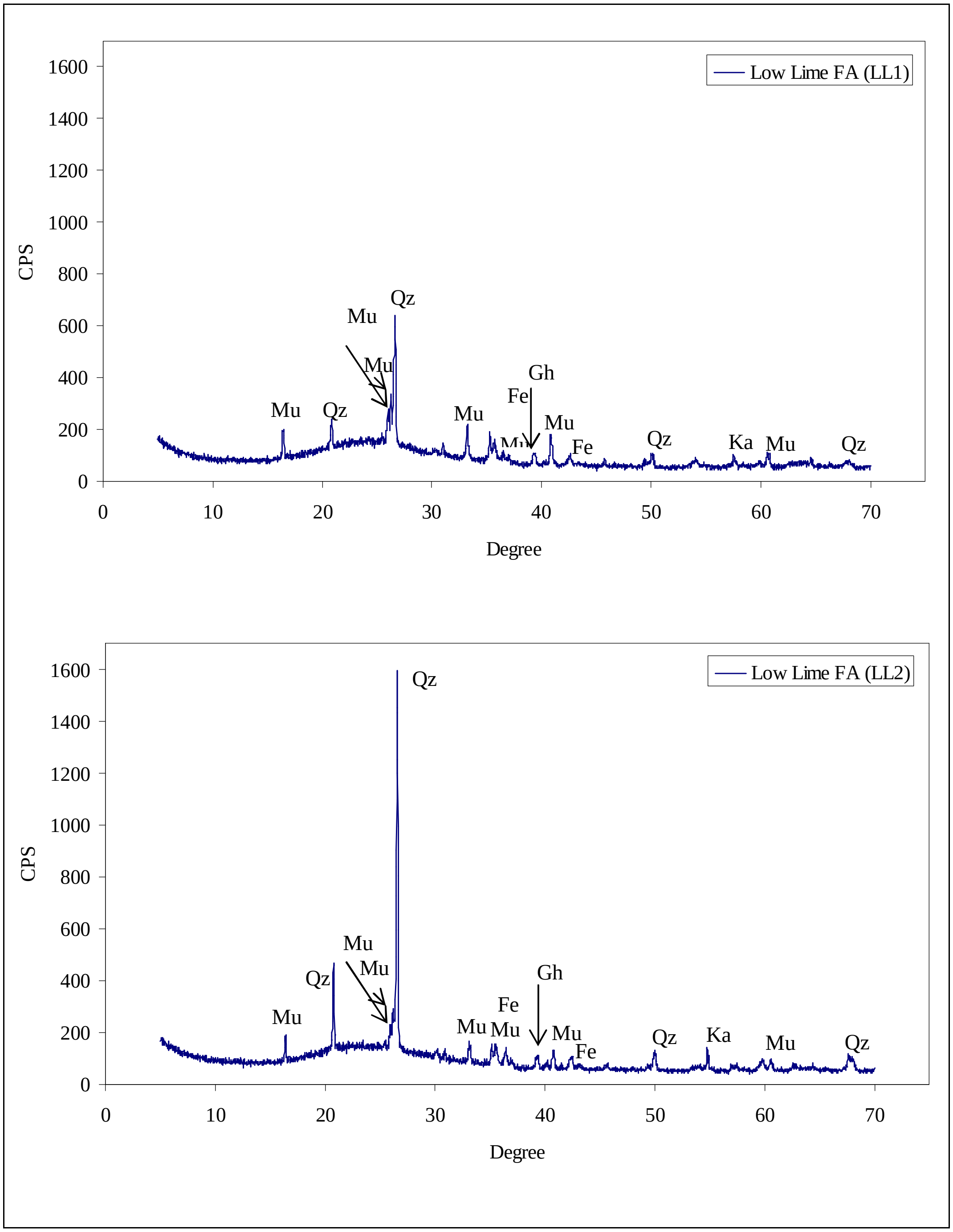



Figure 3.1 X-Ray Diffractograms of Low Lime Fly Ashes (LL1 and LL2)

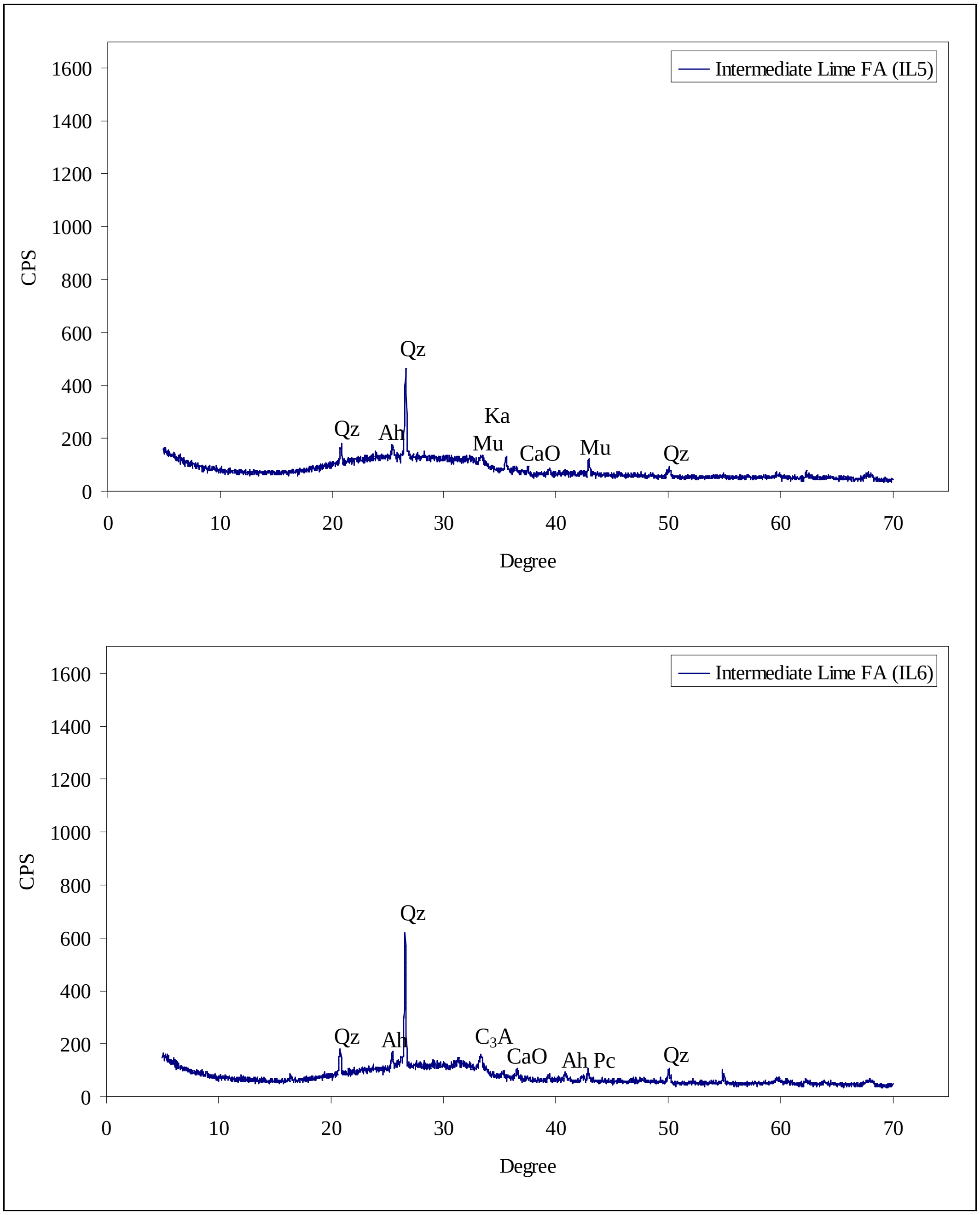



Figure 3.2 X-Ray Diffractograms of Intermediate Lime Fly Ashes (IL5 and IL6)

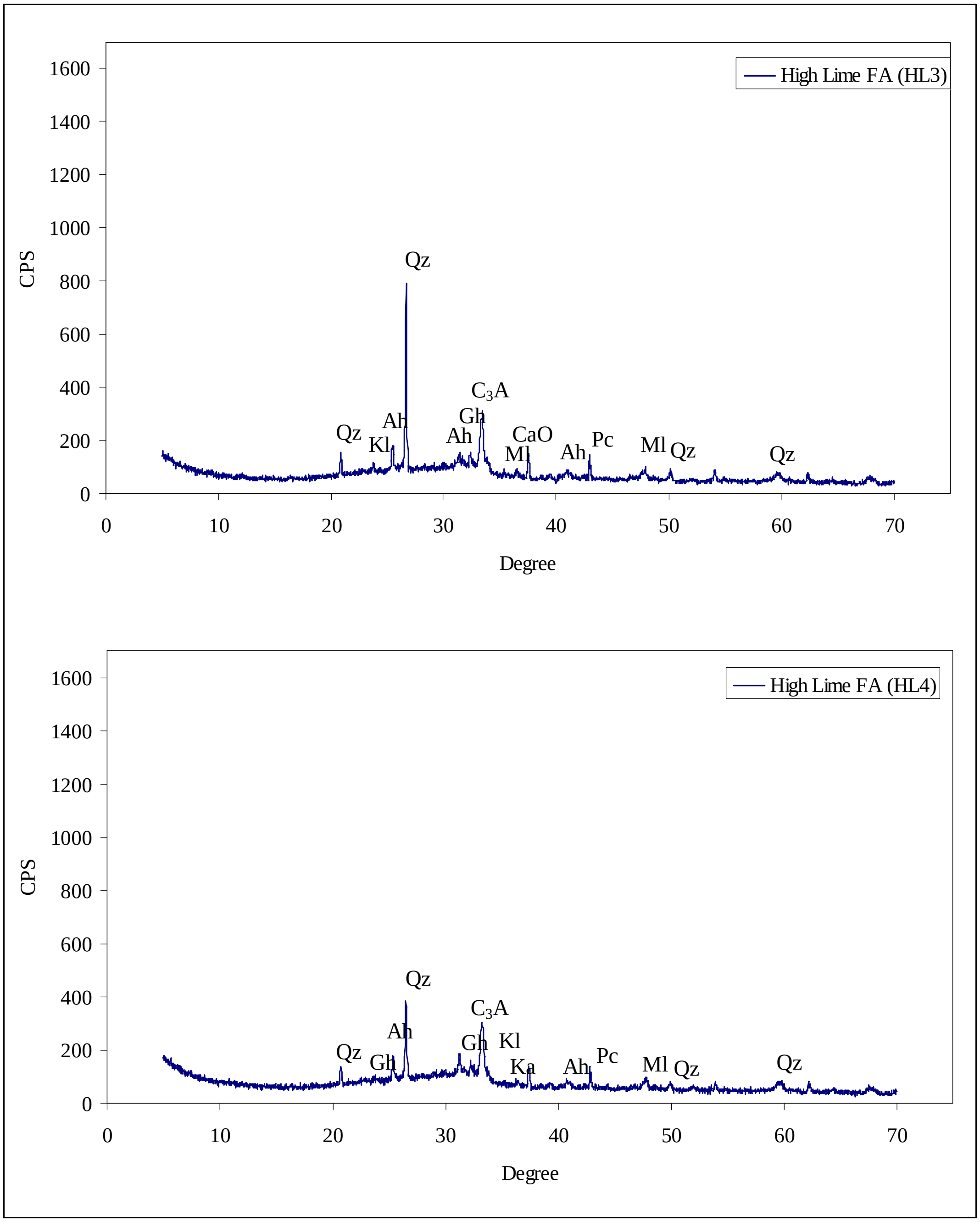



Figure 3.3 X-Ray Diffractograms of High Lime Fly Ashes (HL3 and HL4)

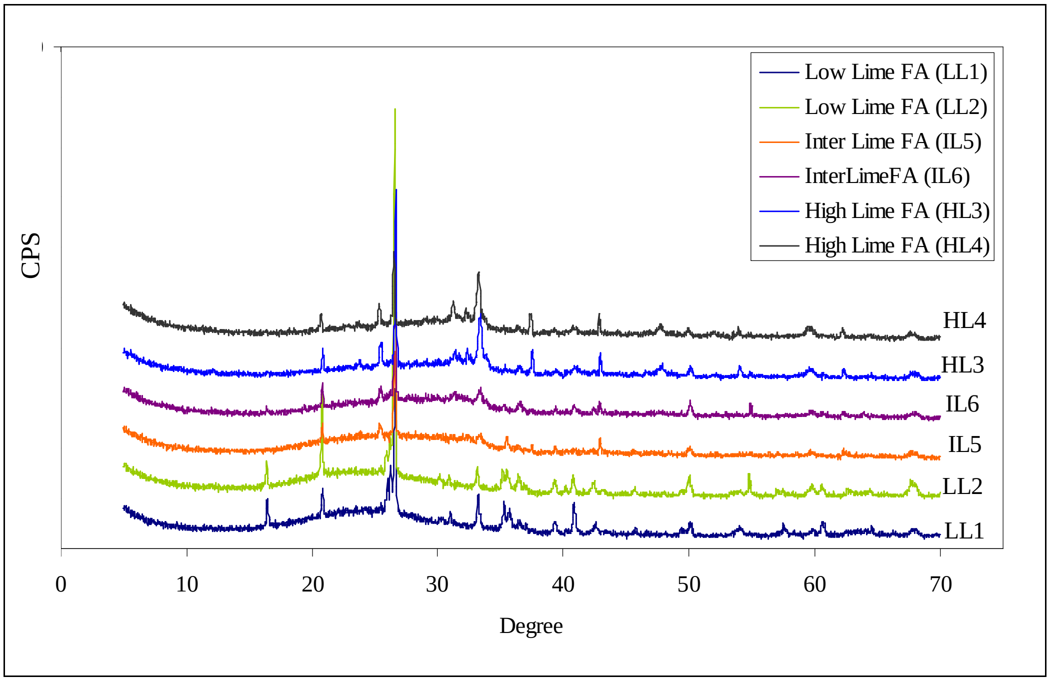



Figure 3.4 Overlay of XRD Patterns of Six Fly Ashes

## 3.4 Mixture Proportions- Standard and Modified ASTM C 1260 and ASTM C 1567 Tests

Table 3.10 presents the mixture proportions for the standard and modified mortar bar tests. The ASTM C 1567 tests include the use of SCMs-fly ash and slag, while the ASTM C 1260 tests are for plain cement-aggregate combinations. In all the mortar bar tests, the cementitious material to aggregate ratio by weight was maintained at 1: 2.25 and the aggregates were graded as per the ASTM C 1260 and ASTM C 1567 specifications. The mixture proportions presented in table 3.11 are for a batch of 4 mortar bars.

## 3.5 Mixture Proportions- Modified ASTM C 1293 Tests

Table 3.11 presents the mixture proportions for the modified (Type-1) and modified (Type-2) ASTM C 1293 tests. The cement replacement by fly ash and slag were on a weight basis and the water/cementitious materials ratio was kept constant at 0.435. Reagent grade sodium hydroxide was added in the mixes made using high alkali cement to raise the total alkali content of the cement to 1.25% of the total weight of cement.

Table 3.10 Mixture Proportions for Standard and Modified ASTM C 1260 and ASTM C 1567 Tests.

| Materials | Control ASTM C 1260 (Std. and Modified) | ASTM C 1567 (Std. and Modified) | | | | |
|---|---|---|---|---|---|---|
| | | FLY ASH | | | SLAG | |
| | | 15% | 25% | 35% | 40% | 50% |
| Cement, g | 500 | 425 | 375 | 325 | 300 | 250 |
| Fly Ash, g | 0 | 75 | 125 | 175 | 0 | 0 |
| Slag, g | 0 | 0 | 0 | 0 | 200 | 250 |
| Water, g | 235 | 235 | 235 | 235 | 235 | 235 |
| Aggregates, g | 1125 | 1125 | 1125 | 1125 | 1125 | 1125 |
| w/cm | 0.47 | 0.47 | 0.47 | 0.47 | 0.47 | 0.47 |

Table 3.11 Mix design for C 1293 tests made with high alkali cement

| Materials (kg/$m^3$) | Spratt | NM | Spratt | NM | IL |
|---|---|---|---|---|---|
| Cement, kg | 25% Fly Ash mix – 315<br>35% Fly Ash mix- 273 | | 40% Slag mix- 252<br>50% Slag mix- 210 | | |
| Fly Ash (25%), kg | 105 | 105 | 0 | 0 | 0 |
| Fly Ash (35%), kg | 147 | 147 | 0 | 0 | 0 |
| Slag (40%), kg | 0 | 0 | 168 | 168 | 168 |
| Slag (50%), kg | 0 | 0 | 210 | 210 | NA |
| Fine agg, kg | 678 | 623 | 678 | 623 | 669 |
| Coarse agg (SSD), kg | 1103 | 1113 | 1103 | 1113 | 1118 |
| Water, kg | 182.7 | 182.7 | 182.7 | 182.7 | 182.7 |
| NaOH addition, kg/ $m^3$ | 25% Fly Ash mix – 1.71<br>35% Fly Ash mix- 1.48 | | 40% Slag mix- 1.37<br>50% Slag mix- 1.14 | | |
| Water/Cementitious | 0.435 | 0.435 | 0.435 | 0.435 | 0.435 |
| Agg./Cementitious | 4.23 | 4.11 | 4.23 | 4.11 | 4.20 |
| Density, kg/$m^3$ | 2383.54 | 2338.90 | 2383.54 | 2338.90 | 2389.53 |

## 3.6 Test Matrix – Mortar Tests

Table 3.12 presents the primary test matrix for the mortar bar and concrete prism tests. Table 3.13 presents the test matrix for mortar bar tests for cement-fly ash combinations with selected aggregates and selected cement replacement by fly ashes. SP, SD, NM, NC and IL represent the five aggregates used in this study and its presence in a particular cell of the table indicates a combination of that aggregate at a particular cement replacement level and with a particular fly ash. Table 3.14 presents the test matrix for mortar bar tests for cement-slag-aggregate combinations at selected cement replacement levels by slag.

Table 3.12 Primary Test Matrix for Mortar Bar and Concrete Prism Tests

| | Test | Soak Solution | Temp. in °C | Aggregate Type | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | IL | SP | NM | NC | SD |
| Mortar Bar Tests | ASTM C 1260 | 1N NaOH | 80 | X | X | X | X | X |
| | Modified ASTM C 1260 | KAc | 80 | X | X | X | X | X |
| | ASTM C 1567 | 1N NaOH | 80 | X | X | X | X | X |
| | Modified ASTM C 1567 | KAc | 80 | X | X | X | X | X |
| Concrete Prism Tests | Modified ASTM C 1293 (1) | 1N NaOH | 38 | X | X | X | X | X |
| | Modified ASTM C 1293 (2) | KAc | 38 | X | X | X | | |

Table 3.13 Test Matrix for Standard and Modified ASTM C 1260 and ASTM C 1567 Tests with Fly Ashes

| **Fly Ash** | **% CaO** | **% Cement Replacement** | | |
|---|---|---|---|---|
| | | **15%** | **25%** | **35%** |
| **LL1** | 1.27 | | SP | |
| **LL2** | 1.34 | | SP | |
| **LL3** | **3.35** | SP, NC, NM, SD | SP, NC, NM, SD, IL | SP, NC, NM, SD |
| **LL4** | 7.31 | | SP | |
| **LL5** | 7.49 | | SP | |
| **IL1** | 10.33 | | SP | |
| **IL2** | 10.45 | | SP | |
| **IL3** | 10.56 | | SP | |
| **IL4** | 12.25 | | SP | |
| **IL5** | **15.63** | SP, NC, NM, SD | SP, NC, NM, SD, IL | SP, NC, NM, SD |
| **IL6** | 18.94 | | SP | |
| **HL1** | 22.85 | | SP | |
| **HL2** | 27.47 | | SP | |
| **HL3** | **27.5** | SP, NC, NM, SD | SP, NC, NM, SD, IL | SP, NC, NM, SD |
| **HL4** | 29.85 | | SP | |

Table 3.14 Test Matrix for Standard and Modified ASTM C 1567 Tests with Slag

| **Slag** | **% Cement Replacement** | |
|---|---|---|
| | **40%** | **50%** |
| **Grade 120** | SP, NC, NM, SD, IL | SP, NC, NM, SD |

## 3.7 Test Matrix – Concrete Tests

Table 3.15 and 3.16 shows the test matrix for the concrete prism tests with three representative fly ashes and one slag respectively.

Table 3.15 Test Matrix for Modified ASTM C 1293 Tests with Fly Ashes

| **Fly Ash Name** | **% CaO** | **% Cement Replacement** | | |
|---|---|---|---|---|
| | | **15%** | **25%** | **35%** |
| **LL3** | 3.35 | - | SP, NM | SP, NM |
| **IL5** | 15.63 | - | SP, NM | SP, NM |
| **HL3** | 27.5 | - | SP, NM | SP, NM |

Table 3.16 Test Matrix for Modified ASTM C 1293 Tests with Slag

| **Slag** | **% Cement Replacement** | |
|---|---|---|
| | **40%** | **50%** |
| **Grade 120** | SP, NM and IL | SP, NM |

# CHAPTER IV RESULTS AND DISCUSSION

## 4.1 General

This chapter presents the results of the various tests described in chapter 3 and interprets the results by providing a discussion based on the theories and mechanisms hypothesized and/or confirmed. A statistical analysis of the results is conducted to understand the correlations between the various tests to help understand the factors that determine the mitigation potential of fly ash and slag in cement-aggregate combinations exposed to potassium acetate deicer.

## 4.2 Results of Standard and Modified ASTM C 1260 Tests

To evaluate the potential of fly ash and slag as ASR mitigation measures the results of the tests conducted on mortar bars made using fly ash and slag are compared with those of mortar bars without any mitigation measure (Control). The results of the standard and modified ASTM C 1260 tests are referred to as 'Control' test results and are used to compare with the results of standard and modified ASTMC 1567 tests.

Figure 4.1(a) and 4.1(b) shows the results of control mortar bars in the standard (1N NaOH) and modified (Potassium acetate) ASTM C 1260 test for all the five aggregates (four reactive and one non-reactive). The results of the four reactive aggregates (NM Rhyolite, SP Limestone, SD Quartzite and NC Argillite) in these tests are from a doctoral research conducted at Clemson University (Sompura 2006).

From the results shown in figure 4.1 and 4.2 it can be seen that the mortar bars made using the four reactive aggregates expanded well above the 0.1% acceptance limit at 14 days test age in both the standard and modified tests.

NM-Rhyolite was the most reactive (1.43%-1N NaOH and 1.56%-Potassium acetate) among the four reactive aggregates, while the IL-Dolomite was found to be non-reactive in both 1N NaOH (0.04%) and potassium acetate deicer (0.05%) solutions. Though the reactive aggregates were identified as 'reactive' in both the tests, it was interesting to note that the expansion of mortar bars containing SP limestone and SD quartzite aggregates showed almost twice the expansion in potassium acetate deicer solution than in 1N NaOH solution. This indicates that the aggregate reactivity, as characterized by the standard ASTM C 1260 test, may not be very representative for certain aggregates where exposure to potassium acetate deicer solutions is to be expected.

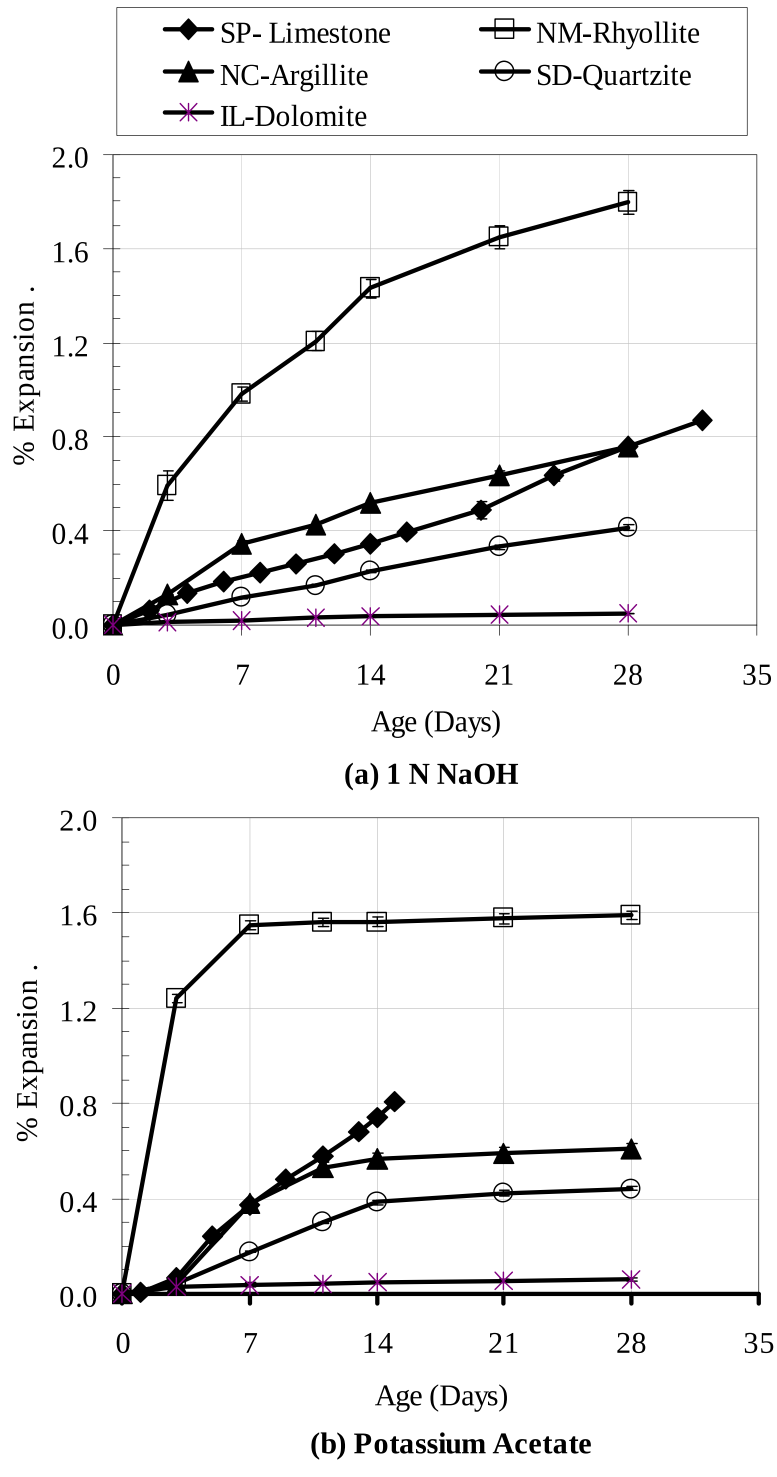



Figure 4.1 Expansions of Control Mortar Bars in the (a) Standard and (b) Modified ASTM C 1260 Tests

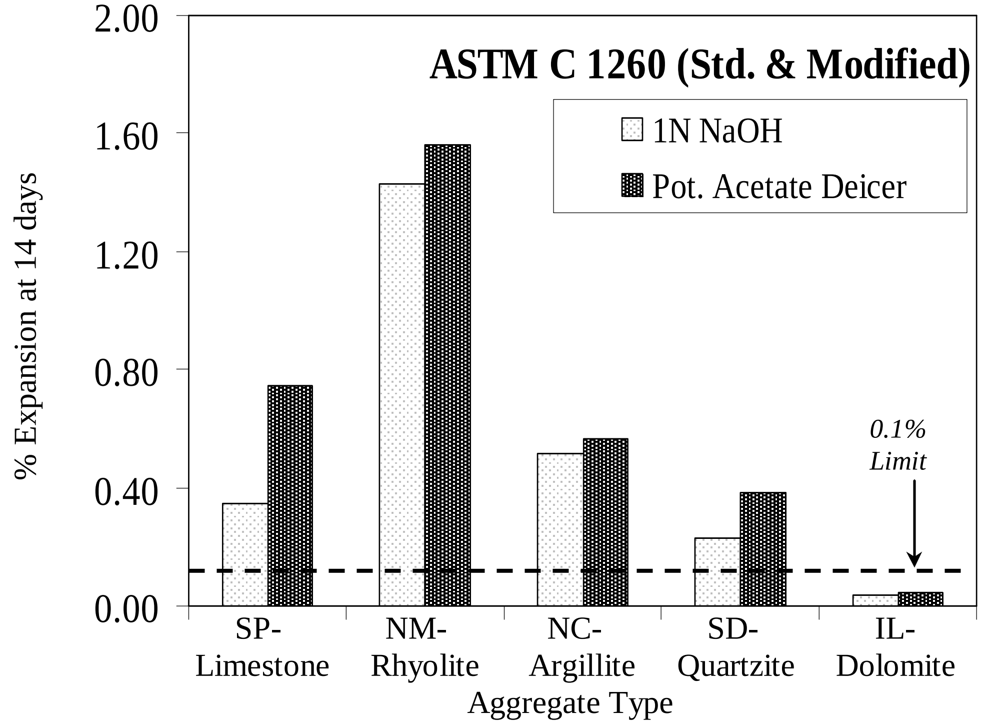



Figure 4.2 14-day Expansion of Control Mortar Bars (i.e. without any mitigation) in the Standard and Modified ASTM C 1260 Tests

## 4.3 Results of Standard and Modified ASTM C 1567 Tests to Investigate the Effectiveness of Fly Ashes

This section presents the results of standard and modified ASTM C 1567 tests in which the effect of fly ash type and fly ash dosage in combination with aggregates exhibiting a range of reactivity are investigated. In addition, SEM and EDX analyses on the samples of the ASTM C 1567 (standard and modified) tests were conducted. Of all the four reactive aggregates Spratt limestone was selected for detailed studies as it has an established history of being ASR reactive in the field and laboratory studies. Also, of the three fly ash dosages (15%, 25% and 35%), detailed studies were conducted for fly ashes at 25% cement replacement level. This selection was based on the premise that 25% fly ash dosage is the most commonly used cement replacement level for fly ashes on concrete airfield pavements and therefore logical to evaluate mortars and concrete samples for their ASR mitigation potential at this dosage when exposed to potassium acetate deicer.

For each of the aggregates, the specific results from the standard and modified ASTM C 1567 tests will be discussed:

- Length-change behavior of mortar bars
- Dynamic modulus of elasticity (DME) of mortar bars at 25% cement replacement
- Microstructure studies of Spratt limestone containing mortar bars with fly ashes at 25% cement replacement level.

In addition, detailed studies on Spratt limestone mortar bars, as listed in the test matrix for ASTM C 1567 tests (Table 3.13), will be presented and discussed.

#### 4.3.1 Spratt Limestone

In the beginning of this study, Spratt limestone was tested with fifteen fly ashes exhibiting a range of chemical composition, lime content in specific, representing three categories –Low lime, Intermediate lime and High lime fly ash. The influence of these fly ashes at one dosage (25%) was investigated to provide a basis for studying other factors such as fly ash dosage and aggregate type.

In the following paragraphs, the results of Spratt mortar bars with fifteen fly ashes at 25% dosage in 1NNaOH and potassium acetate deicer exposure are presented. This is followed by the results and discussion of Spratt mortar bars with three fly ashes at three dosages (15%, 25% and 35%).

Length Change Behavior of Mortar Bars- Spratt with 15 Fly Ashes at 1 Dosage Level

Figure 4.3 shows the expansions of mortar bars made with Spratt limestone and 15 fly ashes having a wide range of chemical compositions, lime (CaO) content in specific, at 25% cement replacement level.

Figure 4.4A and 4.4B shows the expansions of mortar bars made with Spratt aggregate and 15 fly ashes at 14 and 28 days test age respectively. The fly ashes in these figures are represented by their respective lime (%CaO) content so as to observe the influence of lime content on the expansions of mortar bars in both 1N NaOH and potassium acetate deicer exposure. Results will be discussed referring to both these figures to aid discussion.

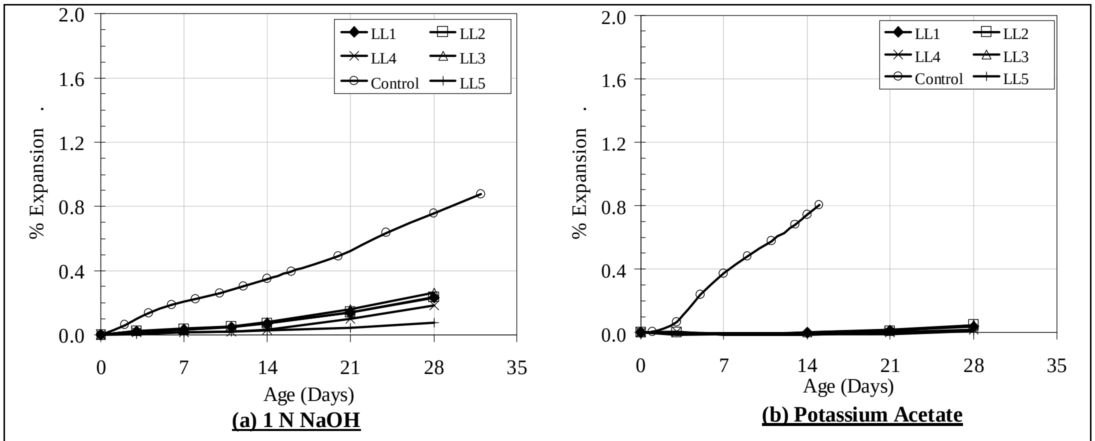
The results are split into three categories based on the lime content of the fly ashes. Figure 4.3A shows the expansion results of mortar bars made with five low lime (%CaO <8.0%) fly ashes at 25% cement replacement in the presence of 1N sodium hydroxide and potassium acetate deicer solutions respectively. The results show that all the five low lime fly ashes were effective in reducing the expansions to below 0.1% at 14 days in both the standard and modified tests. However, mortar bars exposed to sodium hydroxide had expansions that gradually increased over the 28 day test regime and cross the 0.1% limit beyond 14 days. On the contrary, mortar bars exposed to potassium acetate had expansions well below the 0.1% limit up to 28 days. Comparing the effectiveness of five low lime fly ashes among themselves in 1N NaOH exposure, it is found that the expansions increased as the lime content decreased while this trend is reversed for expansions in presence of potassium acetate solution.

Figure 4.3B shows the expansions for mortar bars made with six intermediate lime fly ashes (%CaO 8 to 20%) at 25% cement replacement and comparing them with the control (no fly ash) expansions. The trend of mortar bar expansions in 1N NaOH and potassium acetate deicer was similar to that observed for low lime fly ashes. In 1N NaOH, the expansions remained below 0.1% at 14 days for all but one fly ash (IL6) and then increased above 0.1% beyond 14 days. However, the expansions remained below 0.1% at both 14 and 28 days in potassium acetate exposure (Figure 4.3B and 4.4).

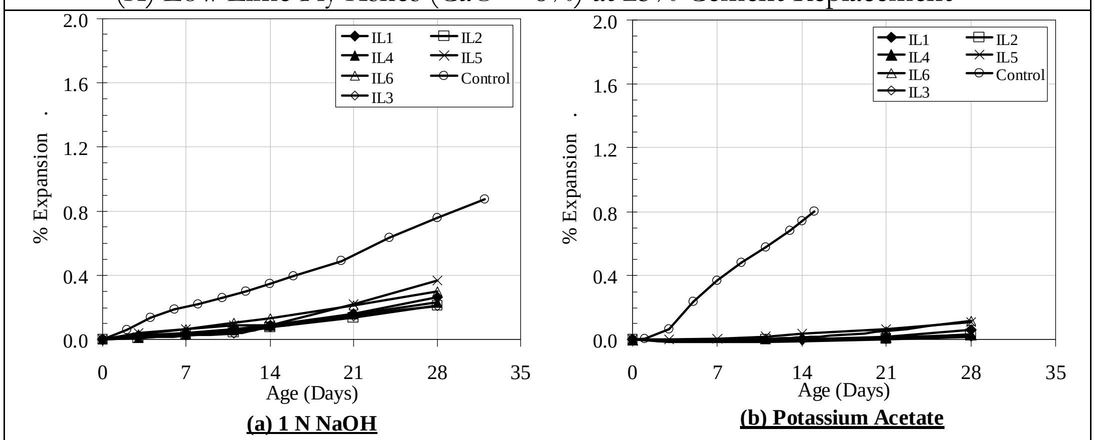
Figure 4.3C shows the expansions for mortar bars made with four high lime fly ashes (%CaO>20%) at 25% cement replacement in 1N NaOH and potassium acetate solution. Mortar bar expansions of none of the four high lime fly ash mixes were below the 0.1% limit at 14 days in the presence of 1N NaOH. Likewise, except for the fly ash (HL1) with the lowest lime content (%CaO- 22.8) among the four fly ashes, mortar bars with the high lime fly ashes expanded more than 0.1% at 14 days.

From figures 4.3 C, 4.4A and 4.4B it is clearly evident that in the presence of potassium acetate there is a dramatic increase in the expansions of mortar bars with fly ashes having lime content greater than 23%. The expansions of mortar bars with fly ashes HL2 (%CaO- 27.47), HL3 (%CaO- 27.5) and HL4 (%CaO- 29.85) were almost similar but considerably higher than HL1 (%CaO- 22.85).

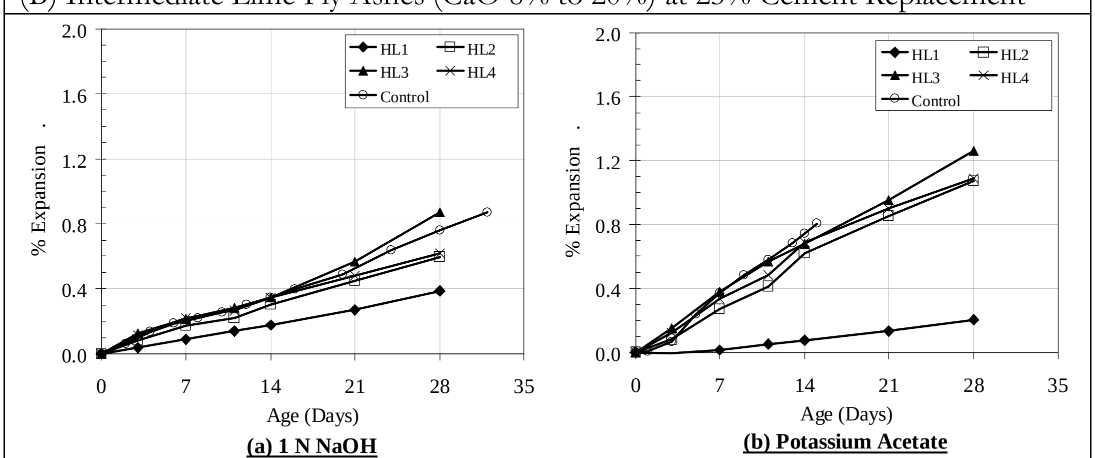
It was noted that up to a fly ash lime content of 22.85%, the 14 and 28 day mortar bar expansions in 1N NaOH were higher than those in potassium acetate. However, for fly ashes with lime content greater than 23%, the mortar bar expansions in potassium acetate were almost twice the expansions in 1N NaOH.




(A) Low Lime Fly Ashes (CaO < 8%) at 25% Cement Replacement




(B) Intermediate Lime Fly Ashes (CaO 8% to 20%) at 25% Cement Replacement




(C) High Lime Fly Ashes (CaO > 20%) at 25% Cement Replacement

Figure 4.3 Expansions of Mortar Bars Containing Spratt Limestone Aggregate in Standard and Modified ASTM C 1567 Tests with 15 fly ashes at 25% Fly Ash Dosage

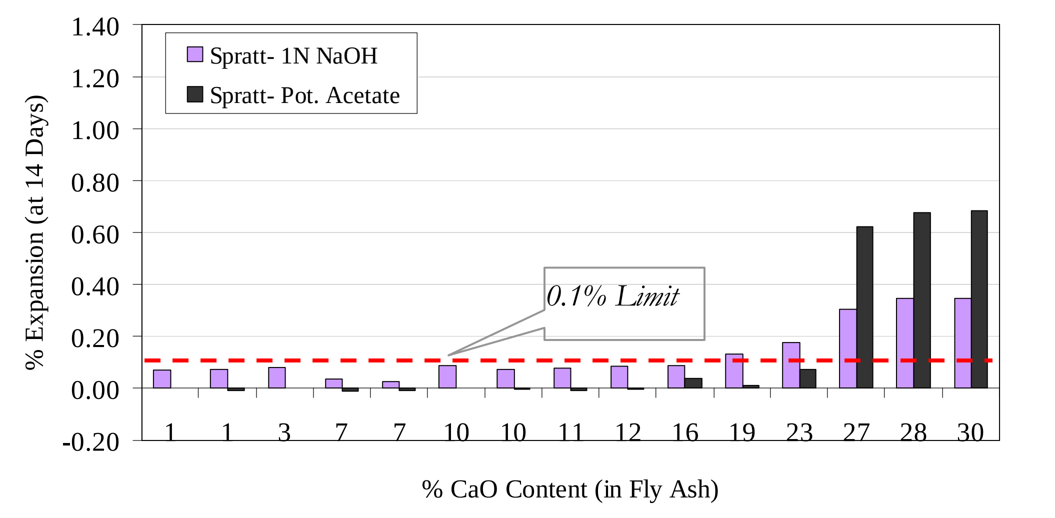



Figure 4.4(A) 14 Day Expansions of Mortar Bars with 15 Fly Ashes

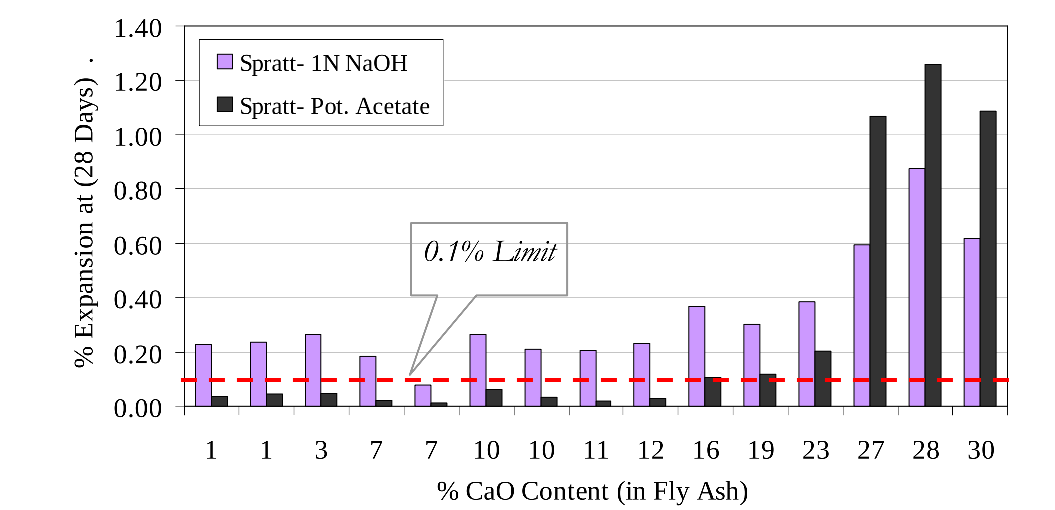



Figure 4.4(B) 28 Day Expansions of Mortar Bars with 15 Fly Ashes

Figure 4.4 14 and 28 day Expansions of Mortar Bars Containing Spratt Limestone Aggregate in Standard and Modified ASTM C 1567 Tests with 15 Fly Ashes at 25% Cement Replacement

Length Change Behavior of Mortar Bars- Spratt with 3 Fly Ashes at 3 Dosage Levels

The results of the length change behavior of mortar bars are presented in a manner to show two aspects of ASR mitigation using fly ash; (a) influence of the fly ash lime content on mortar bar expansions and (b) influence of fly ash dosage on the mortar bar expansions. Figure 4.5 shows the expansion of mortar bars with three fly ashes selected from the fifteen fly ashes previously discussed. Each of the three fly ashes represents one of the three categories- Low lime, Intermediate lime and High lime fly ash based on their lime content (CaO -3.35%, 15.63% and 27.5%). These three fly ashes were used at three dosages (A-15%, B-25%, C-35%) in combination with Spratt in 1N NaOH and potassium acetate soak solution. The results of these three fly ashes at 25% dosage are also presented in figure 4.3 and 4.4. The results are compared with the no fly ash (control) mortar bar results.

Based on the results shown in figure 4.5, certain trends are evident with respect to the fly ash dosage, fly ash lime content, the exposure conditions (1N NaOH or potassium Acetate deicer solution) and the rate of expansion.

In the standard ASTM C 1567 tests (1N NaOH solution exposure), it was observed that as the fly ash dosage increases, the 14 day expansions decrease or match the control expansions regardless of the fly ash type. 25% and 35% fly ash dosages of low lime and intermediate lime contents were found to be adequate to suppress the expansions below the 0.1% acceptance limit at 14 days. However, this trend could be misleading given the fact that in certain cases the expansions increased above the 0.1% just after 14 days. High lime fly ash was ineffective in mitigating the expansions at all the three dosages in 1N NaOH exposure and the expansions were either comparable or more than the control samples.

Results of the mortar bars soaked in potassium acetate deicer solution showed similar trends in mortar bar expansions as discussed for those in 1N NaOH. Low lime and intermediate lime fly ashes at 25% and 35% dosages were very effective in reducing the mortar bar expansions in potassium acetate deicer exposure. Unlike the increased expansions observed beyond 14 and 28 days by the mortar bars containing low and intermediate lime fly ash in 1N NaOH solution, the expansions remained below 0.1% up to 56 days in potassium acetate.

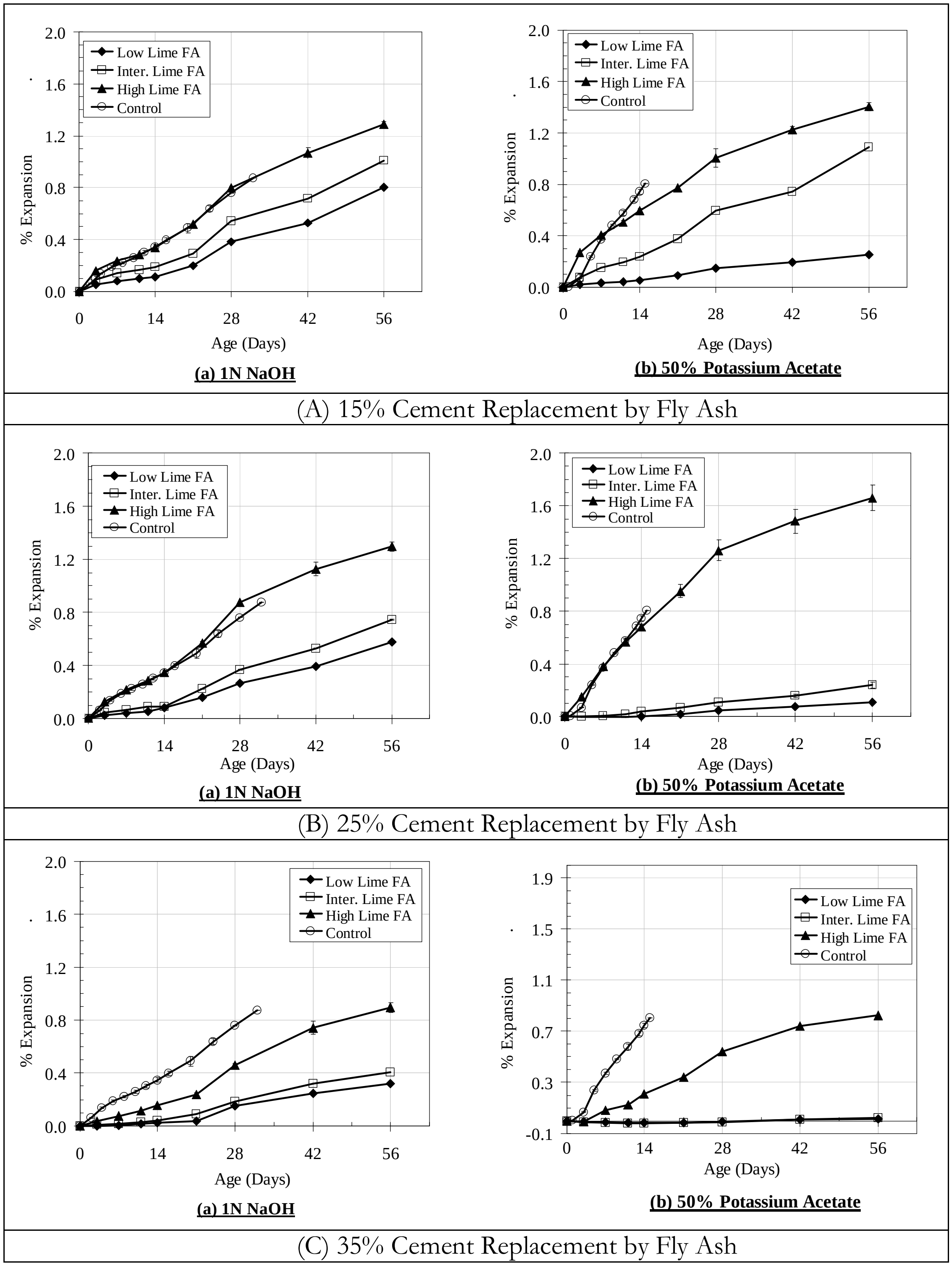



Figure 4.5 Expansions of Mortar Bars Containing Spratt Limestone Aggregate in Standard and Modified ASTM C 1567 Tests with Fly Ash at 15%, 25% and 35%

Influence of ASTM C 1567 Test regime on Dynamic Modulus of Elasticity (DME)

Figure 4.6 shows the changes in the dynamic modulus of elasticity (DME) of mortar containing Spratt limestone aggregate and three fly ashes of varied lime contents at 25% cement replacement in the Standard (1N NaOH) and modified (potassium acetate) ASTM C 1567 Tests.

Based on the results shown in figure 4.6, it is evident that there is a pronounced drop in the DME of the mortar bars with high lime fly ash in both 1N NaOH and potassium acetate exposure. This drop in DME corroborates the increase in the expansions of these bars by indicating a loss of physical integrity of the cement aggregate matrix. Contrary to the significant drop in DME of high lime fly ash containing mortar bars, the mortar bars containing low and intermediate lime fly ashes had a much smaller drop and the trend lines appear to follow a plateau. Comparing the change in DME of mortar bars in 1N NaOH and potassium acetate exposure, the low and intermediate lime fly ash mortar bars performed better in potassium acetate with a negligible drop in DME over the test regime. These results are consistent with the length change measurements of the mortar bars and there appears to be a very good correlation between the DME and expansion of mortar bars.

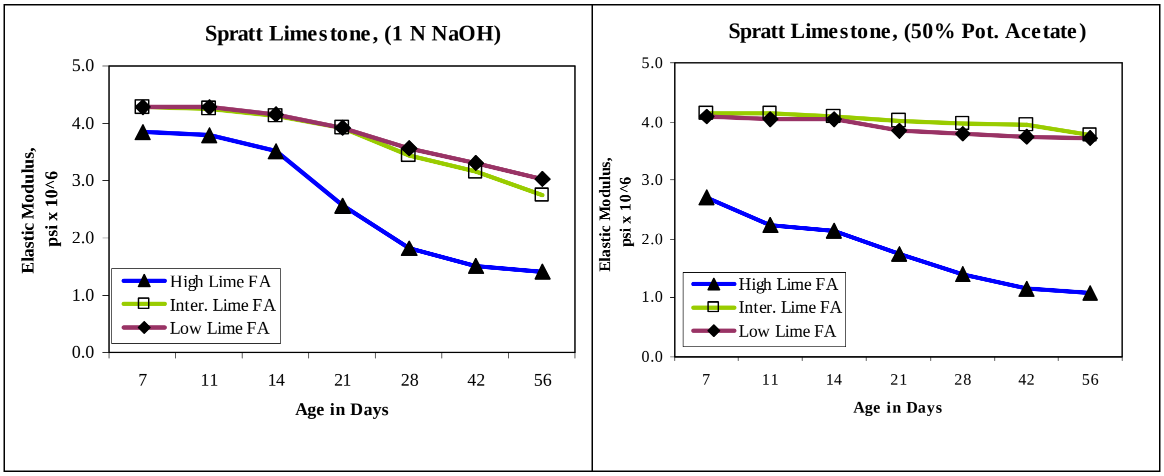



Figure 4.6 Changes in Dynamic Modulus of Elasticity of Spratt Limestone Mortar Bars in Standard and Modified ASTM C 1567 Tests with 25% Fly Ash Dosage

### 4.3.2 Microstructure Studies- Spratt Limestone Mortar Bars

This section presents the results from microstructure investigation and a discussion based on the studies conducted on the mortar bar samples from the standard and modified ASTM C 1567 tests.

#### Spratt Limestone Mortar Bars Containing Low Lime Fly Ash

*1N NaOH Soak Solution*

Figure 4.7 shows the visual images of mortar bars along with a low magnification SEM image of Spratt limestone mortar bar with low-lime fly ash (LL3) soaked in 1N NaOH for 28 days. Figure 4.8 shows a detailed image of the area surrounding the aggregate.

The absence of any significant cracking in the cement matrix or on the surface of the mortar bars is consistent with the length change results presented in figures 4.3 and 4.4 that indicate a significant mitigation in expansion in the mortar bars at 25% low lime fly ash dosage. Minor hairline cracks were observed in the cement paste but were limited to the paste itself and no cracks were observed within the aggregate particles or originating from them into the mortar matrix. The paste surrounding the aggregate particle was particularly rich in sodium as seen in the EDX spectrum shown in figure 4.8.

*Potassium Acetate (KAc) Soak Solution*

Figure 4.9 shows the visual images and SEM micrographs of Spratt limestone mortar bars with low-lime fly ash (LL3) soaked in potassium acetate for 28 days. Figure 4.10 and 4.11 shows the magnified images of regions surrounding an aggregate particle and fly ash grain.

These figures indicate that the cement paste is marked by a cluster of fine cracks through out the sample but there were no cracks observed that emanated from a single aggregate particle and ran across the cement paste. Understandably, there were no signs of physical cracking seen on the surface of the mortar bars. The absence of such severe cracks corroborates the mitigation of expansion by low lime fly ash in the modified ASTM C 1567 test. The EDX spectra of a spot in the cement paste indicate a rich presence of potassium ions that might have infused from the potassium acetate soak solution. The EDX spectra of a fly ash grain and the cement paste surrounding it are shown in figure 4.10 and 4.11. The darker regions within the cement paste were found to be rich in silica and are assumed to be formed due to decalcification of the cement paste.

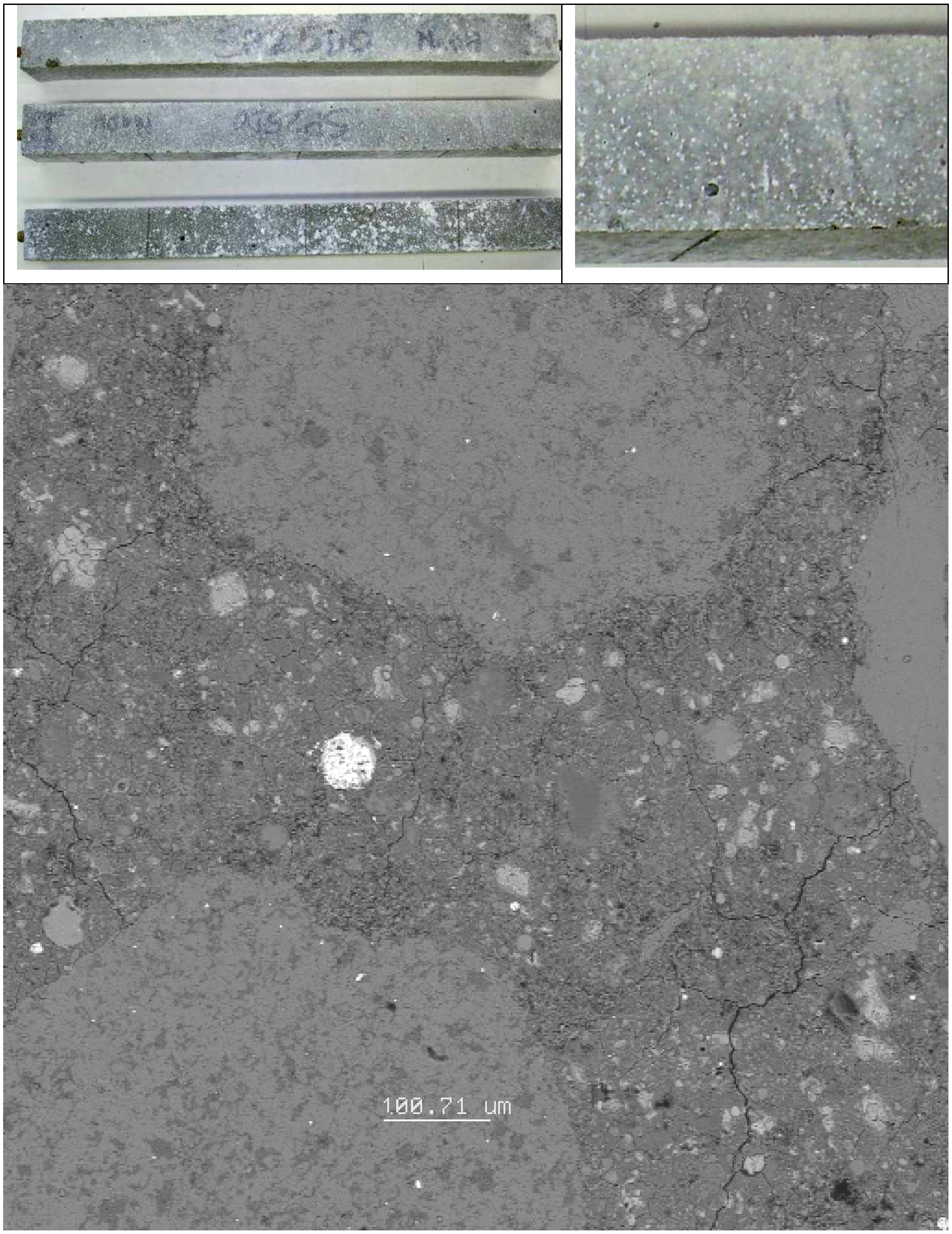



Figure 4.7 Visual Image and Low Magnification SEM Micrograph of Spratt-Limestone Mortar Bar Containing Low-Lime Fly Ash Exposed to 1N NaOH Soak Solution for 28 days

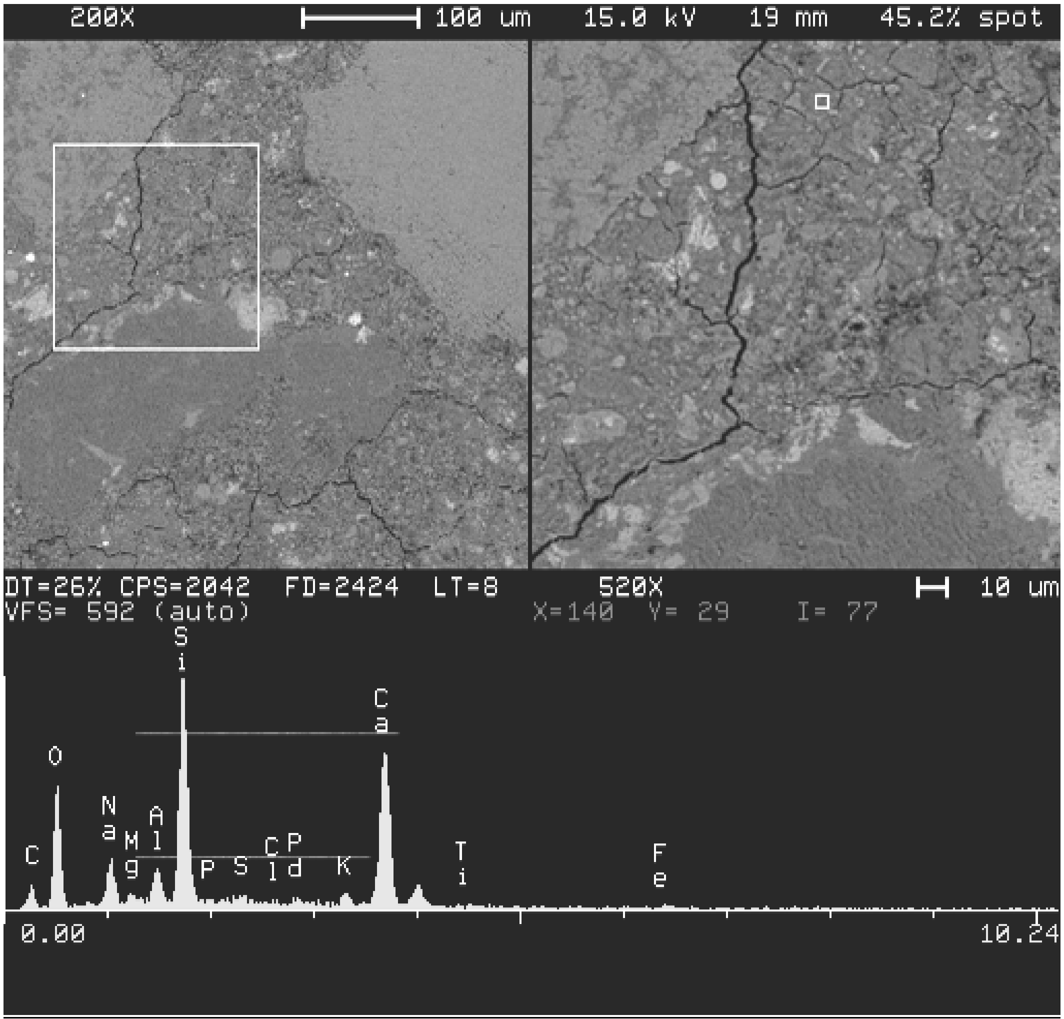



Figure 4.8 SEM Micrograph of Spratt Limestone Mortar Bar Containing Low-Lime Fly Ash At 25% Dosage Level Soaked in 1N NaOH Soak Solution for 28 Days

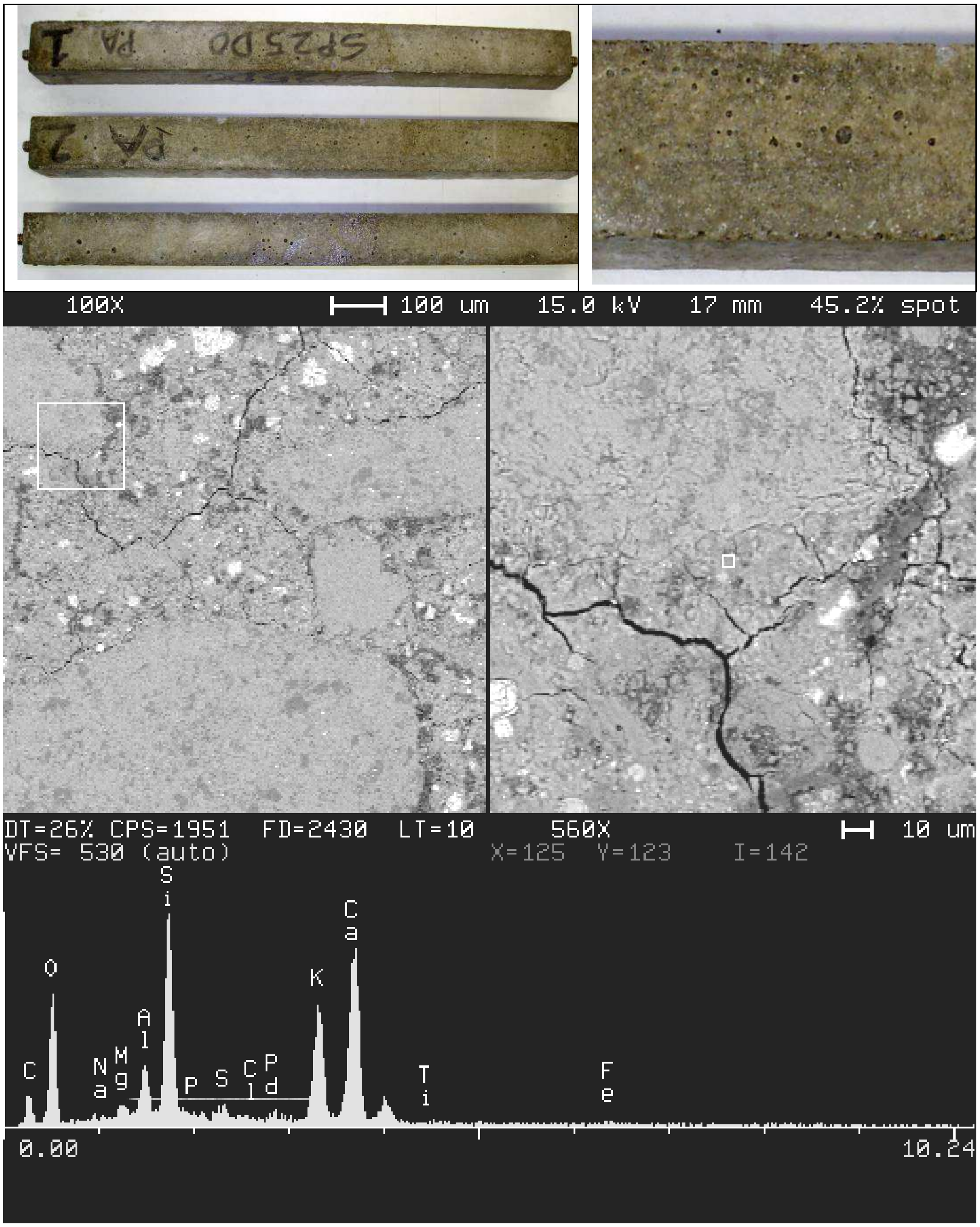



Figure 4.9 Visual Images and SEM Micrograph of Spratt Limestone Mortar Bar Containing Low-Lime Fly Ash At 25% Dosage Level Soaked In KAc Deicer Soak Solution for 28 Days

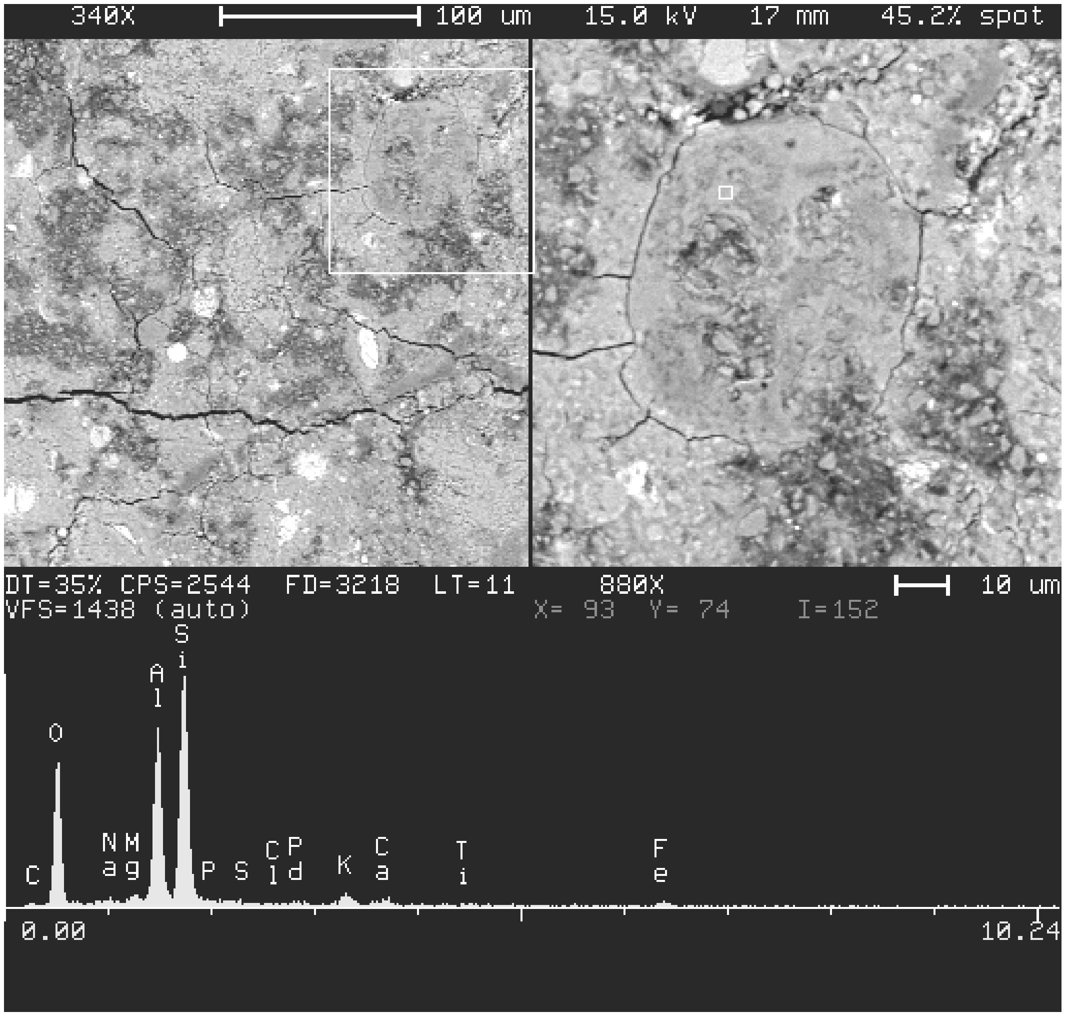



Figure 4.10 SEM Micrograph of Spratt Limestone Mortar Bar Containing Low-Lime Fly Ash At 25% Dosage Level Soaked in KAc Deicer Soak Solution for 28 Days (EDX Spot on Fly Ash Grain

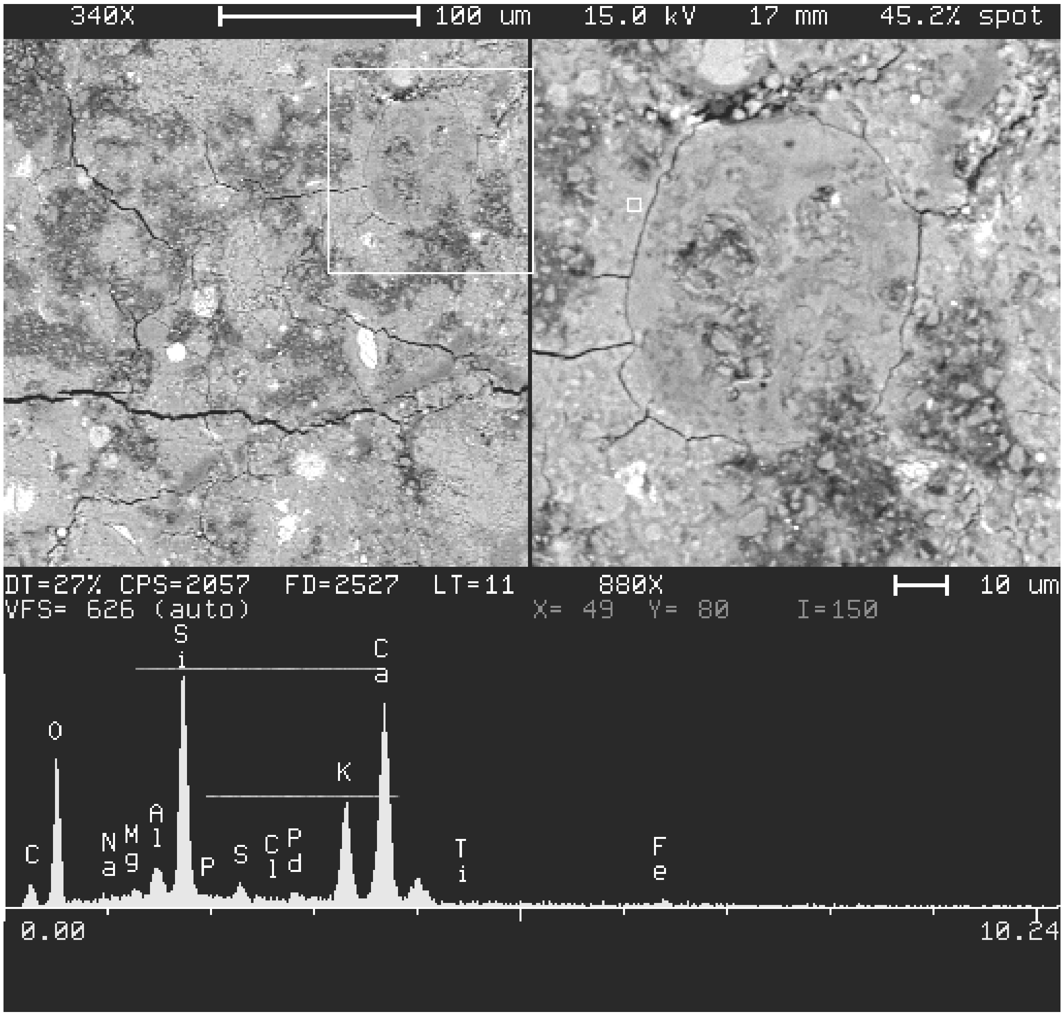



Figure 4.11 SEM Micrograph of Spratt Limestone Mortar Bar Containing Low-Lime Fly Ash At 25% Dosage Level Soaked in KAc Deicer Soak Solution for 28 Days (EDX Spot on Paste Adjacent to Fly Ash Grain

<u>Spratt Limestone Mortar Bars Containing Intermediate Lime Fly Ash</u>

*1N NaOH*

Figure 4.12 and 4.13 shows the visual images and SEM micrographs of Spratt limestone mortar bar with intermediate lime fly ash (IL5) soaked in 1N NaOH for 28 days.

The SEM micrographs indicate a significant amount of cracking in the cement paste. However, similar to the observation made in low lime fly ash-1N NaOH samples, no severe cracks were observed within the aggregate particles. The EDX spectra- shown in figure 4.12 and 4.13 of the paste surrounding the aggregate particles were found to be rich in sodium.

*Potassium Acetate (KAc)*

Figure 4.14 and 4.15 shows the microstructure of Spratt limestone mortar bars containing intermediate lime fly ash exposed to potassium acetate deicer solution for 28 days.

It is evident from the SEM micrographs that the intensity of cracks in the intermediate lime fly ash-cement paste is relatively higher than that observed for low lime fly ash mortar bar samples. The network of cracks is larger and the cracks appear to be wider in comparison to the low lime fly ash samples. However, the cracking is restricted to the aggregate-paste interface only and no significant cracking was observed within the aggregate particles. The cement paste around the aggregate particles was found to be rich in potassium. Minor surfacial cracks could be observed on the mortar bars and provide an evidence of the expansion occurring within them. It was observed in figure 4.15 that though the cracks were restricted to the aggregate-paste interface, some minor cracks were seen near the aggregate periphery within the aggregate.

Though the expansion of mortar bars with intermediate lime fly ash was higher than that of low lime fly ash containing mortar bars, it was significantly lower than the control and also within the 0.1% limit at 14 days.

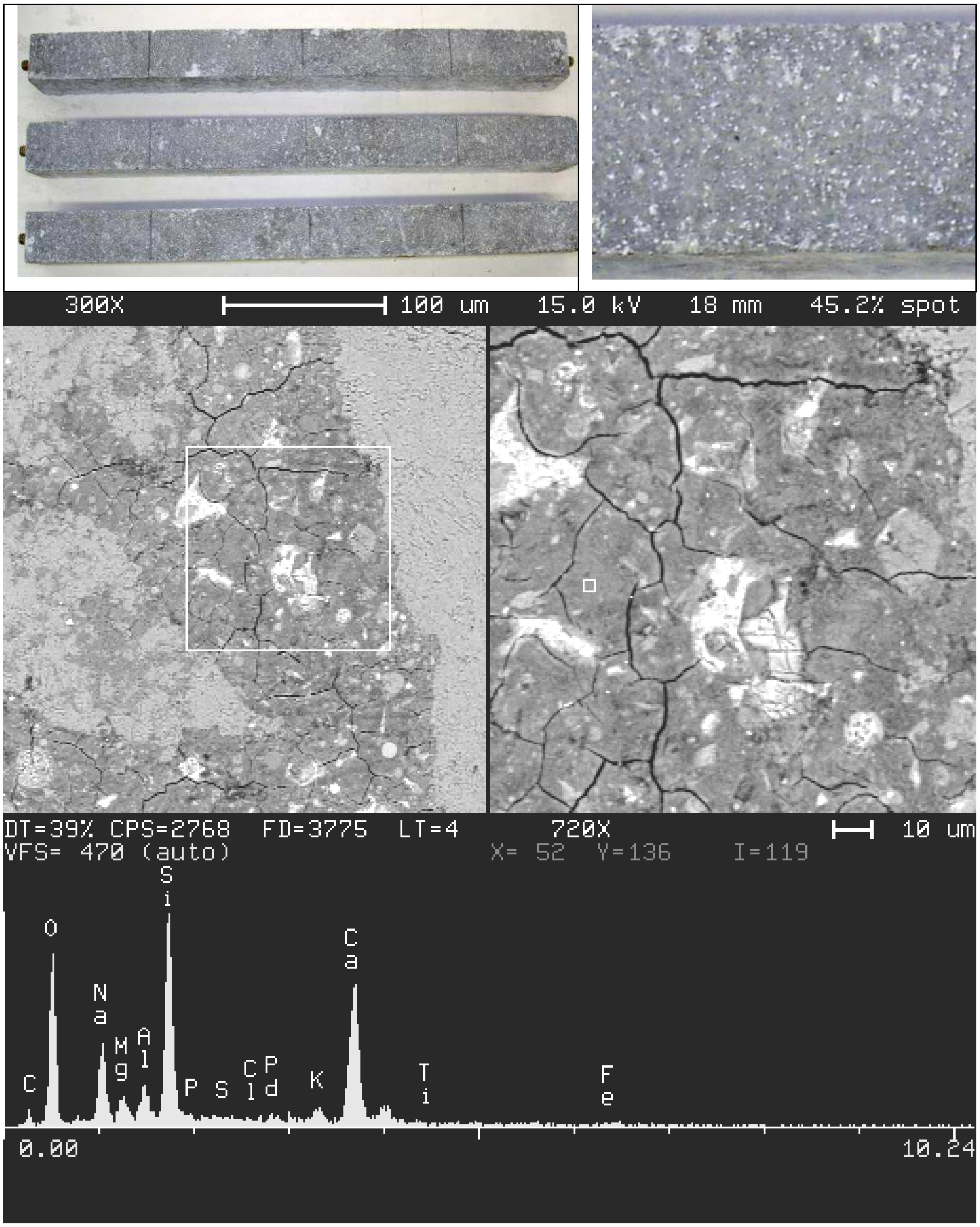



Figure 4.12 Visual Images and SEM Micrographs of Spratt Limestone Mortar Bar Containing Intermediate-Lime Fly Ash At 25% Dosage Level Soaked in 1N NaOH Soak Solution for 28 Days

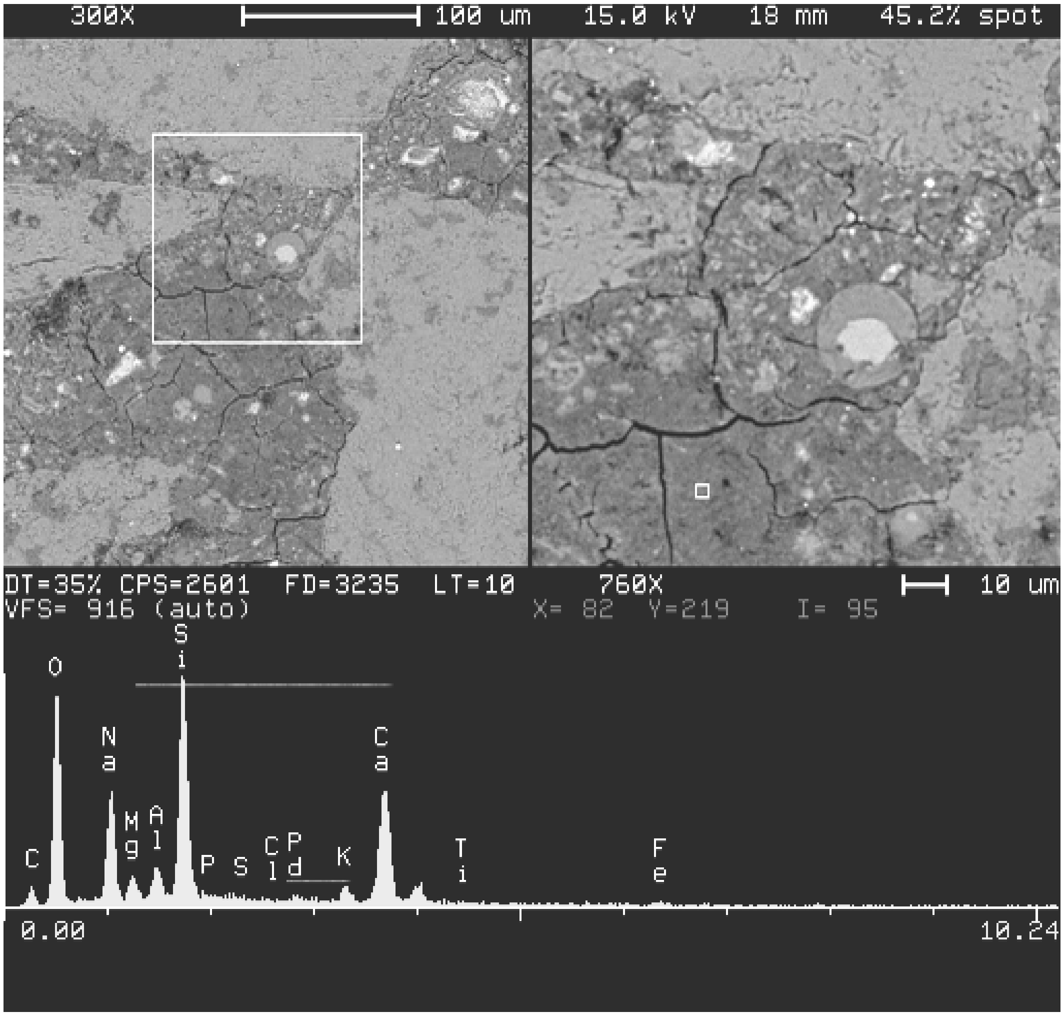



Figure4.13 SEM Micrograph of Spratt Limestone Mortar Bar Containing Intermediate-Lime Fly Ash At 25% Dosage Level Soaked in 1N NaOH Soak Solution for 28 Days

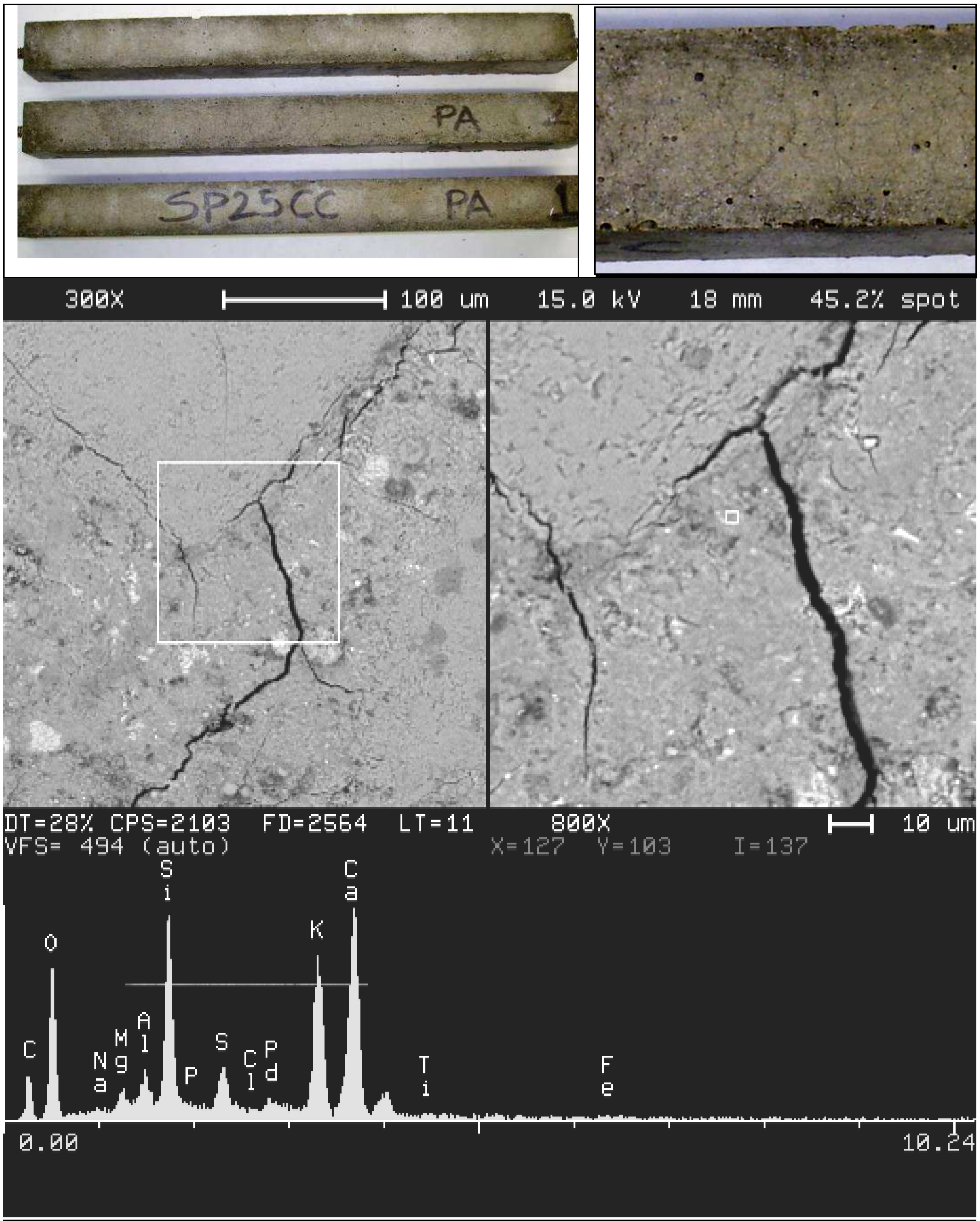



Figure 4.14 SEM Micrograph of Spratt Limestone Mortar Bar Containing Intermediate-Lime Fly Ash At 25% Dosage Level Soaked In KAc Deicer Soak Solution for 28 Days

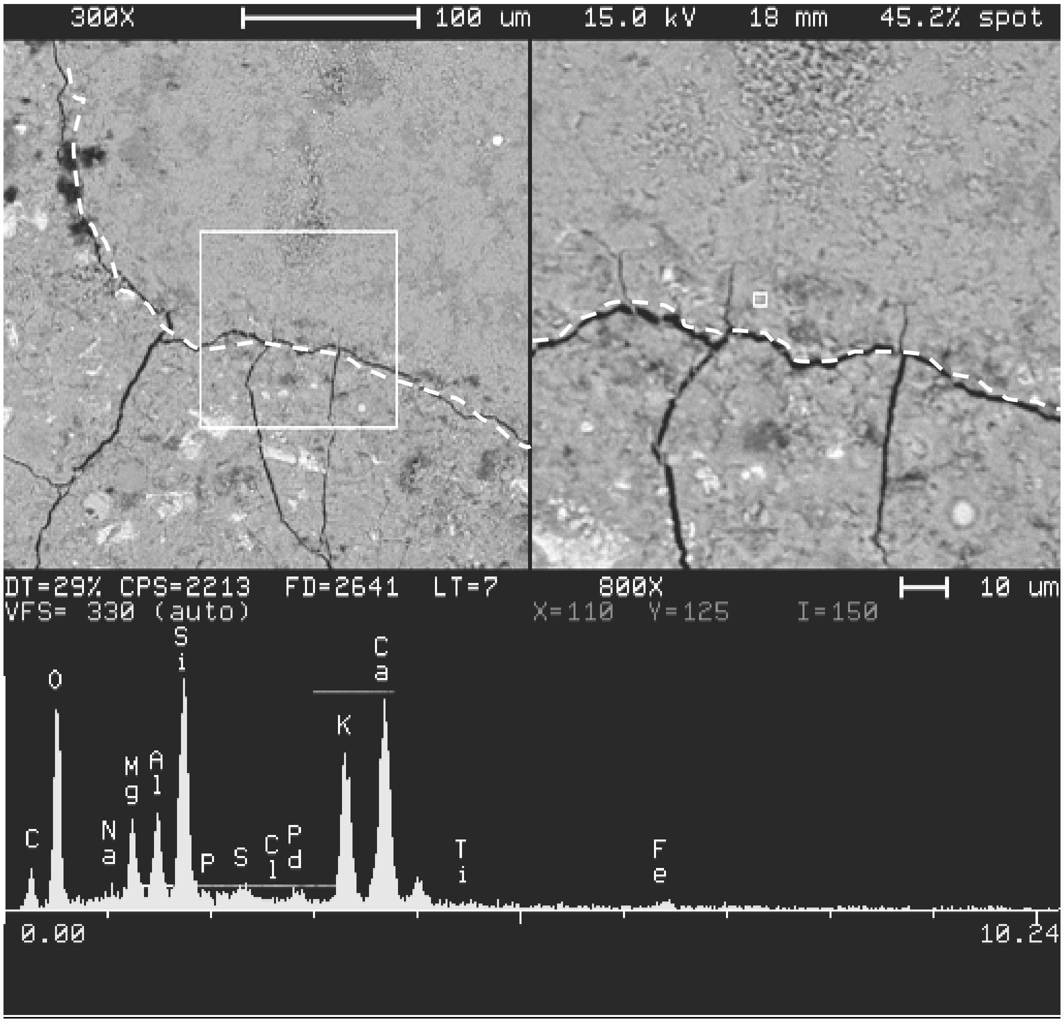



Figure 4.15 SEM Spratt Limestone Mortar Bar Containing Intermediate-Lime Fly Ash At 25% Dosage Level Soaked In KAc Deicer Soak Solution for 28 Days.
The Paste-Aggregate interface is identified with a hashed line

<u>Spratt Limestone Mortar Bars Containing High Lime Fly Ash</u>

*1N NaOH Soak Solution*

Figure 4.16 and 4.17 shows the visual images and SEM micrographs of Spratt limestone mortar bar with high lime fly ash (HL3) soaked in 1N NaOH for 28 days.

The distinguishing feature between the physical distress observed in the cement paste and the aggregate particles among the mortar bar samples containing low, intermediate and high lime fly ash ashes is the occurrence of wide traverse cracks running through the aggregate and continuing in the cement paste. Such cracks are a predominant feature of the mortar bar samples containing high lime fly ash. Figures 4.16 and 4.17 illustrate this point and the high expansions observed in the standard ASTM C 1567 test appear consistent with the signs of physical distress observed in the visual and SEM images.

The SEM images show the presence of ASR gel deposits on the walls of the crack traversing through the aggregate and leading into the cement paste. The EDX spectra of the reaction product conclude that the deposits on the crack walls are ASR gel. The gel formed as a result of the ASR appears to diffuse in the cement paste through the cracks generated by the expansion stresses and as a result, the surrounding paste exhibits desiccation cracks characteristic of the ASR gel.

*Potassium Acetate (KAc) Soak Solution*

Figure 4.18 shows visual images of mortar bars containing Spratt limestone and 25% high lime fly ash exposed to potassium acetate deicer solution. The high expansions of the mortar bars as noted in the results of the modified ASTM C 1567 test were characterized by significant map cracking on the surface that originate from within the mortar matrix as shown in figures 4.19 and 4.20. Another distress characteristic of these mortar bars was the pronounced arching along the length of the mortar bars accompanied by severe cracking on the surface. This arching of mortar bars was more evident in samples soaked in potassium acetate than those exposed to 1N NaOH.

Figure 4.19 shows a region in the mortar bar sample that represents the typical characteristics of distress as observed for high lime fly ash and Spratt limestone combination. Three EDX spots were analyzed to show the interaction of potassium acetate deicer, cement paste and aggregate. It is evident from the EDX that the aggregate particle is influenced by the presence of potassium from the deicer solution. Location A is on the Spratt aggregate particle and the EDX spectra confirms this by showing a high calcite mineral peak. However, on moving towards the aggregate periphery within the aggregate particle, the EDX at location B indicates the presence of minor potassium that has likely migrated into the aggregate from the paste. The EDX spectrum at location C that is in the cement paste and near the aggregate-paste interface shows significant potassium levels that are diffused from the deicer soak solution.

Figure 4.20 shows the presence of a dense ASR gel like reaction product on the periphery of an aggregate particle. The EDX spectra shown at the three locations –paste, interface and aggregate are similar to that seen in figure 4.19 where the levels of potassium follow a decreasing trend on moving from the paste towards the aggregate particle. The hashed lines show a tract of the reaction product that is characteristic of ASR gel.

It is believed that the reaction mechanism in the presence of potassium acetate deicer might be a topical chemical reaction between the aggregate surface and the potassium rich cement paste. This theory is further discussed in section 4.11 while discussing the results of the silica dissolution study.

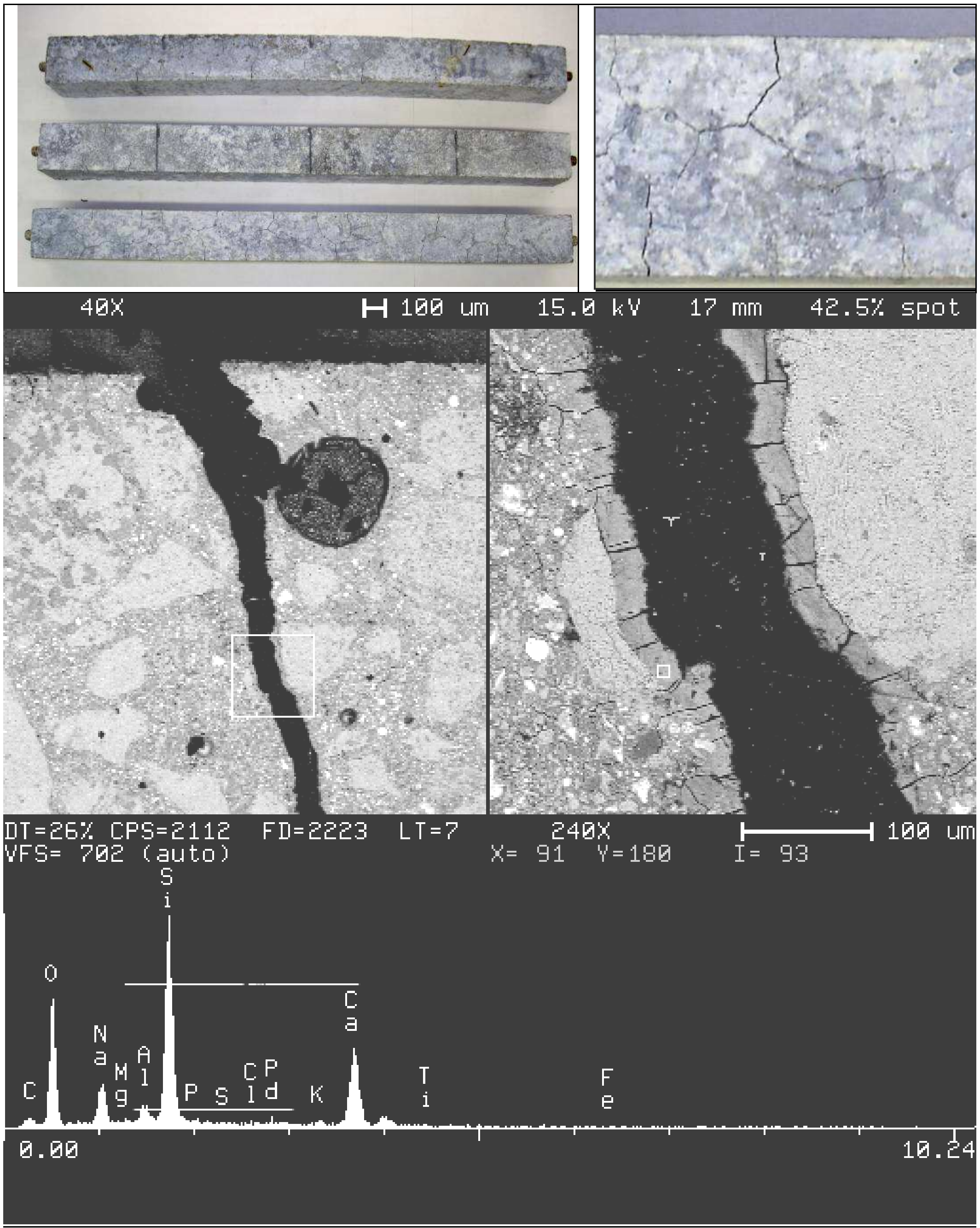



Figure 4.16 SEM Micrograph of Spratt Limestone Mortar Bar Containing High-Lime Fly Ash At 25% Dosage Level Soaked In 1N NaOH Soak Solution for 28 Days

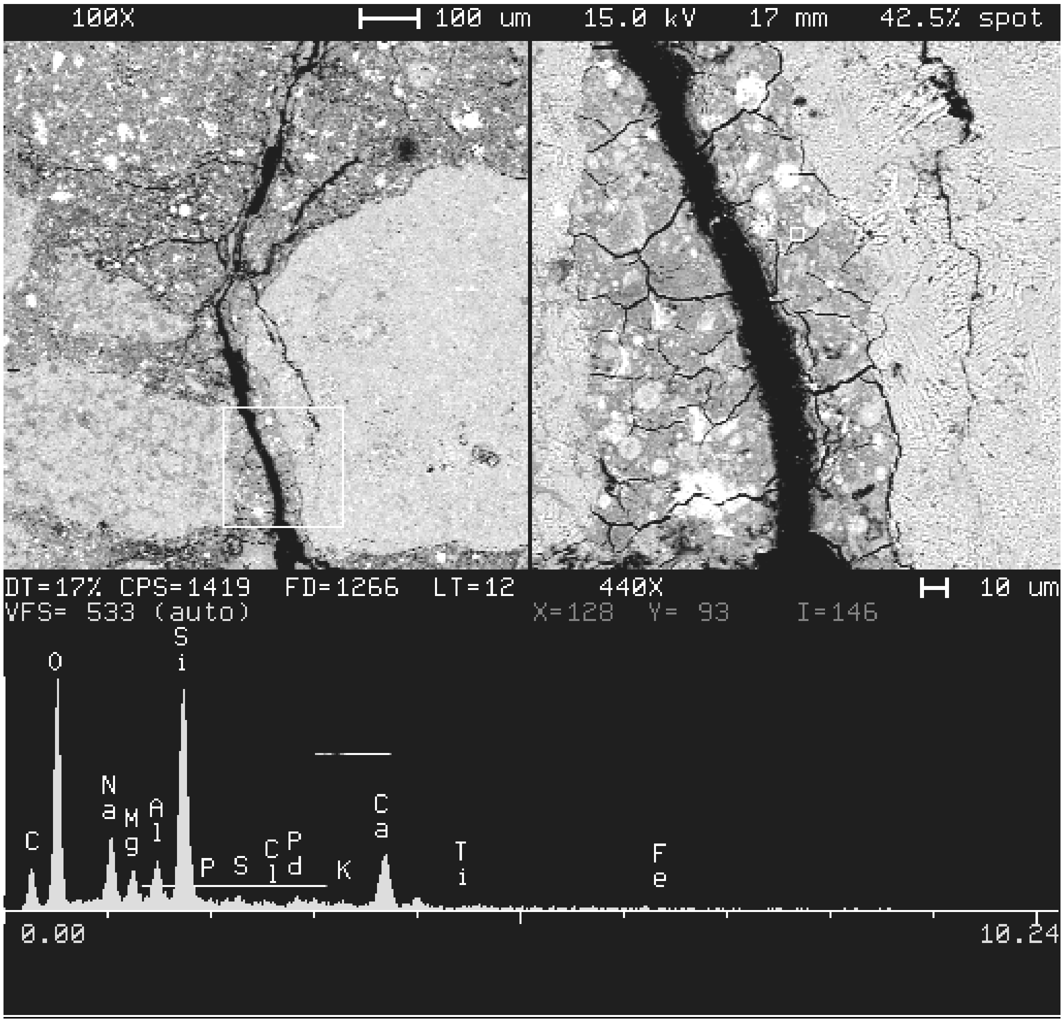



Figure 4.17 SEM Micrograph of Spratt Limestone Mortar Bar Containing High-Lime Fly Ash At 25% Dosage Level Soaked In 1n NaOH Soak Solution for 28 Days.


Figure 4.18 Visual Images of Spratt Limestone Containing Mortar Bars With High Lime Fly Ash at 25% Dosage Level Soaked in KAc Solution for 28 Days.

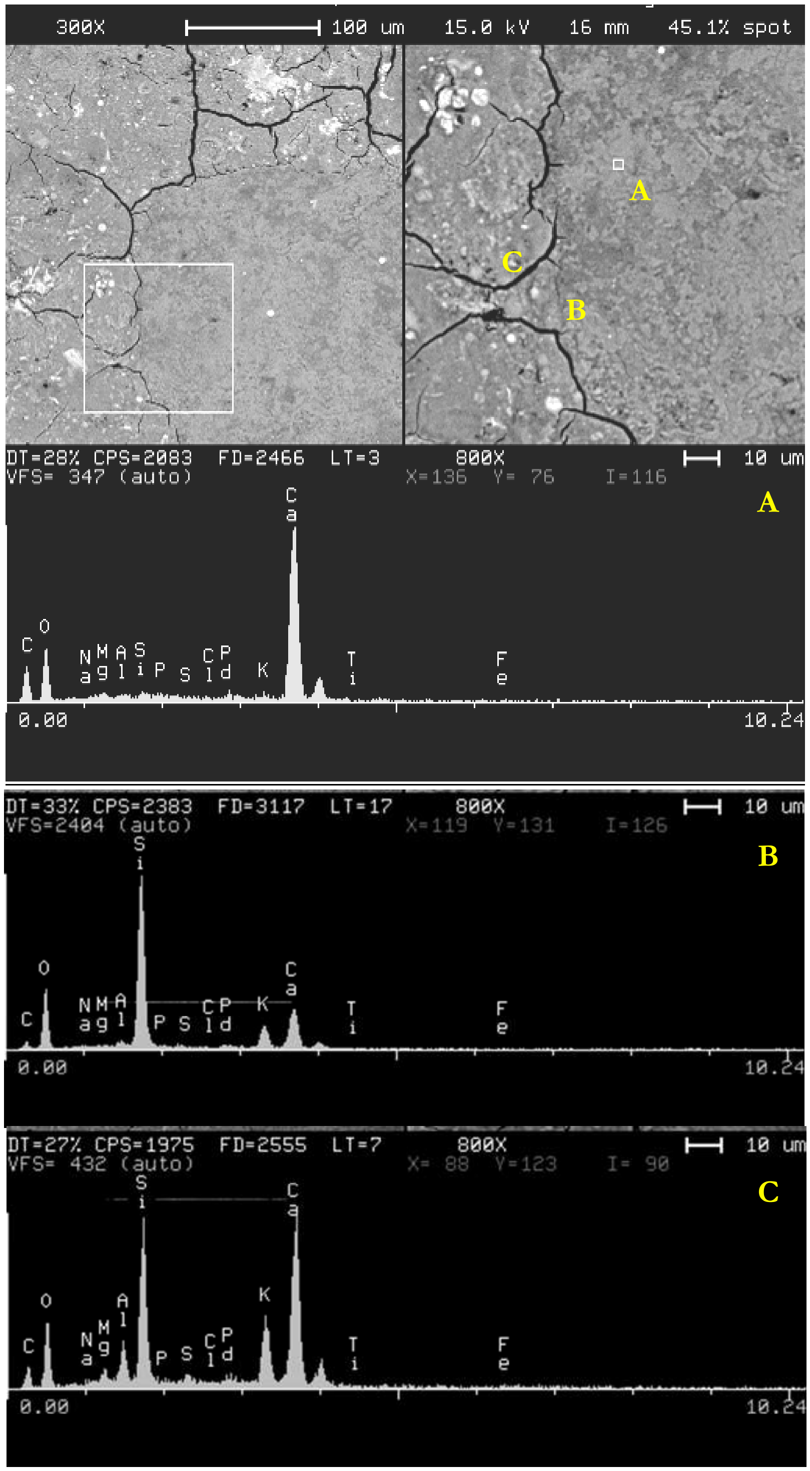



Figure 4.19 SEM Micrograph of Spratt Limestone Mortar Bar Containing High-Lime Fly Ash At 25% Dosage Level Soaked in KAc Deicer Soak Solution for 28 Days (A – Aggregate; B – Interface; C – Paste)

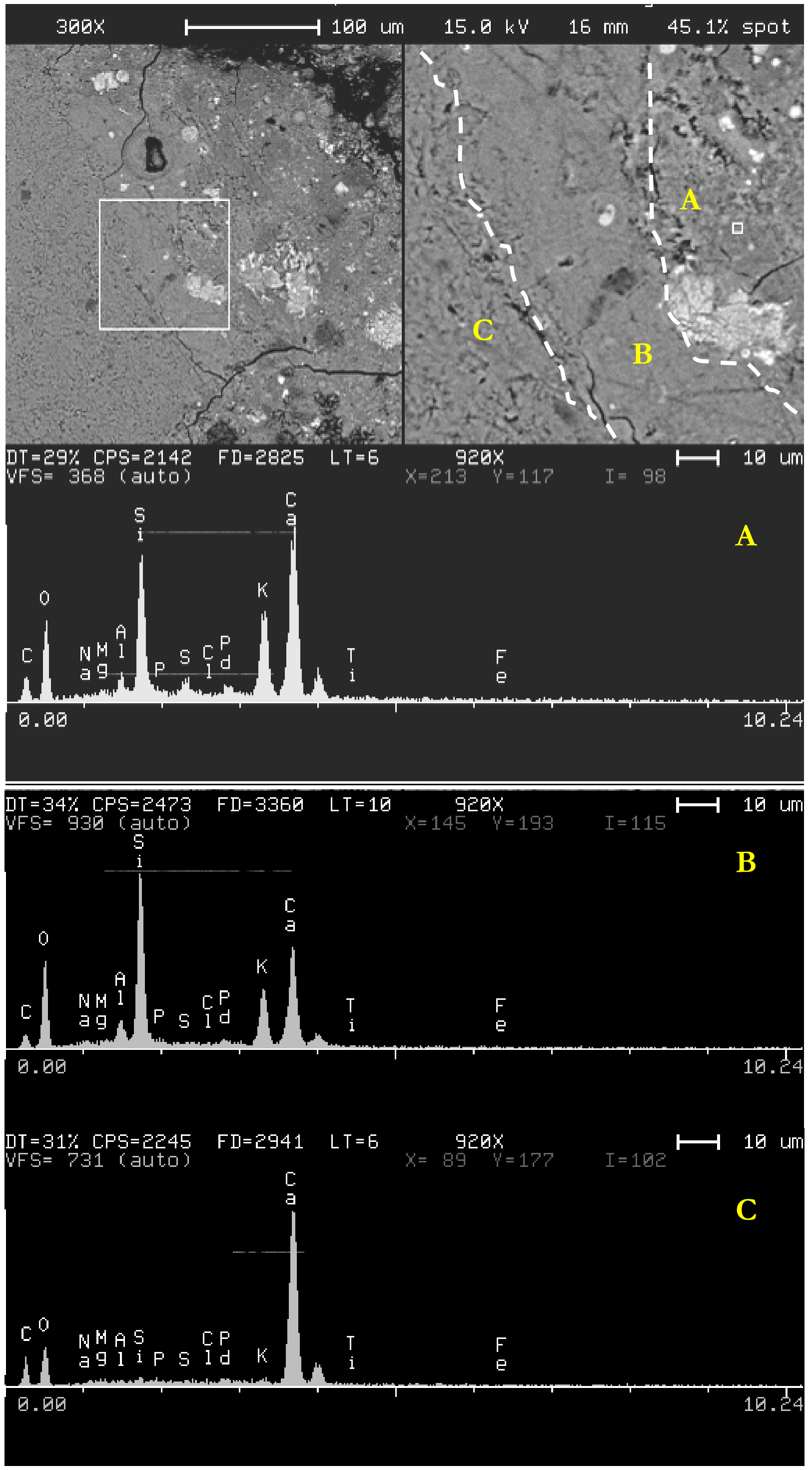



Figure 4.20 SEM Micrographs of Spratt Limestone Mortar Bar Containing High-Lime Fly Ash At 25% Dosage Level Soaked In KAc Deicer Soak Solution for 28 Days (A – Paste; B – Interface; C – Aggregate)

Effectiveness of Fly Ash in Reducing ASR Expansions of Mortar Bars Containing Spratt Limestone

Figure 4.21 shows the percent increase/decrease in the 14 day expansions of the mortar bars containing Spratt limestone aggregate with the three types of fly ashes at 15%, 25% and 35% dosage each. The expansions are compared with the Spratt control (0% fly ash) mortar bar expansion which is bench marked as 0%. A negative percent value indicates a reduction in the expansion and hence being effective.

Based on the values of percent reduction in the expansions for each fly ash at each of the three dosages, it is clearly evident that low lime and inter-mediate lime fly ashes at 25% and 35% are highly effective in reducing the expansions in both 1N NaOH and potassium acetate. However, high lime fly ash becomes effective only at 35% dosage. It should be noted that all the fly ashes irrespective of the dosage used are more effective in reducing the expansions in potassium acetate exposure than in 1N NaOH. Low lime and intermediate lime fly ashes provide similar reduction at both 25% and 35% dosage.

These results should not be seen in isolation and the trend of expansions over the test regime should be observed too. Also, these results do not indicate if the expansions are within 0.1% limit.

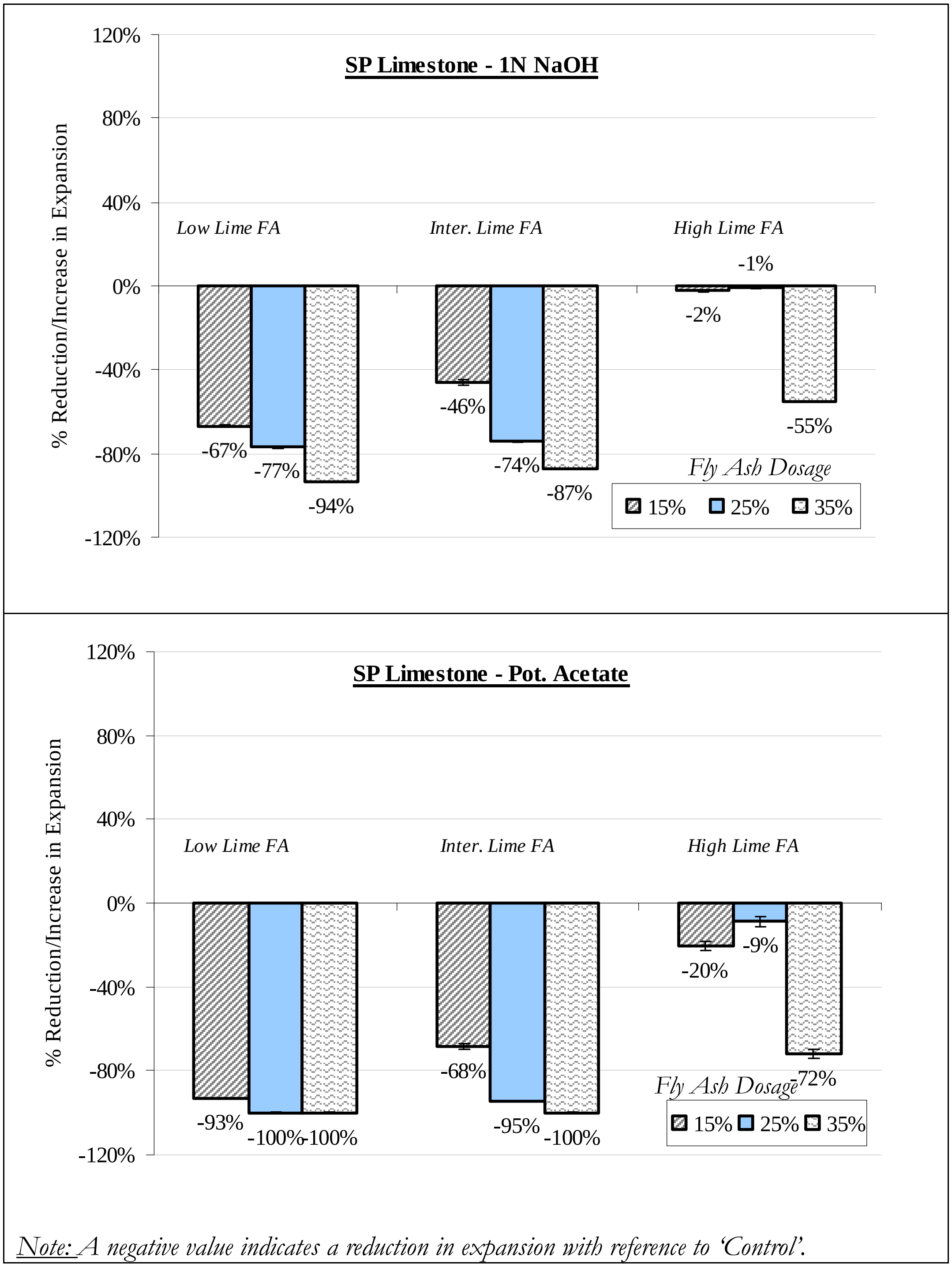



Figure 4.21 Percent Reduction/Increase in the 14 Day Mortar bar Expansions as a Factor of Fly Ash Type and Dosage in 1N NaOH and KAc Exposure with Spratt Limestone as Aggregate.

### 4.3.3 New Mexico Rhyolite

Figure 4.22 shows the expansion of mortar bars containing NM Rhyolite aggregate and fly ash in the presence of 1N NaOH and potassium acetate deicer solutions in the standard and modified ASTM C 1567 tests. The results of these tests are compared with the expansions of the control mortar bar tests.

Results of the standard and modified ASTM C 1567 tests indicate that only low lime fly ash at a dosage of at least 35% is necessary to suppress the mortar bar expansions to below 0.1% at both 14 and 28 days. This can be seen in figures 4.22A, 4.22B and 4.22C. Figure 4.22B show that although at 25% dosage of low lime fly ash the expansions of mortar bars were below 0.1% in both 1N NaOH and potassium acetate, there was a sudden increase in the expansions beyond 14 days. This increase was more pronounced in the case of mortar bars exposed to potassium acetate deicer. Intermediate lime fly ash at 25% dosage was effective in controlling the expansions up to 7 days, after which a sudden jump in the expansions was observed.

Mortar bars with high lime fly ash and NM rhyolite aggregate had the highest expansions (2.13% at 14 days in modified ASTM C 1567) among all the three fly ashes at all the three dosages when exposed to potassium acetate. The expansions were much more in the presence of potassium acetate than in 1N NaOH. In fact at 25% dosage of high lime fly ash the expansions were higher than the control mortar bars (Refer figure 4.22B and 4.24), indicating a deleterious reaction mechanism between the cement, fly ash, aggregate and potassium acetate solution.

Comparing the results of NM aggregate with all the three fly ashes at 15%, 25% and 35% dosages, it appears that low lime fly ash at 35% dosage is more effective in reducing the expansions in mortar bars exposed to potassium acetate deicer solution than in 1N NaOH solution. This trend is similar to what is observed with Spratt limestone aggregate tests.

It was observed that there the results of the expansions of mortar bars at 25% fly ash dosage in potassium acetate had high variability. This was noticeable especially in the case of low and intermediate lime fly ash mortar bars. The reason behind this is believed to be the expansive stresses generated due to the alkali-silica reaction which result in extensive physical damage of the mortar bars in the form of cracking. Once the bars show signs of severe cracking, the expansions of the mortar bars tend to vary from one specimen to the other depending on the extent of cracking. Since, each of the data points forming the trend line represent an average of the expansion values of 3 mortar bars, a high expansion of even one mortar bar among the set of three bars results in a high standard deviation value. This reasoning is justified by looking at the expansion trend for mortar bars with intermediate lime and low lime fly ashes in potassium acetate shown in figure 4.22B. Up to 14 days the mortar bar matrix of cement, fly ash and aggregate was able to contain the expansive stresses inspite of the cracking. However, beyond 14 days one of the three mortar bars had higher expansions compared to the other two leading to the variability in the results and longer error bars on the data points.

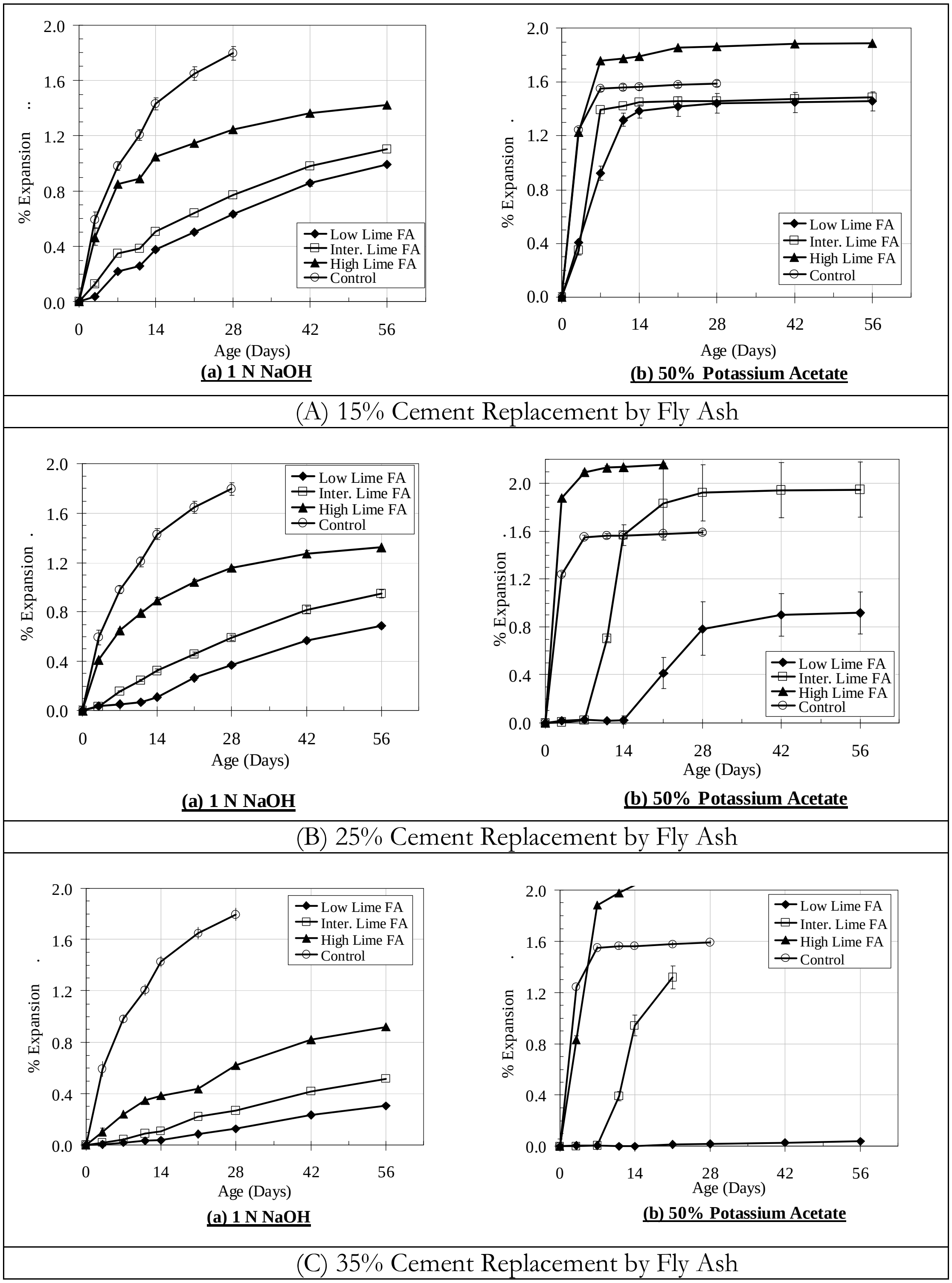



Figure 4.22 Expansions of Mortar Bars Containing New Mexico Rhyolite Aggregate in Standard and Modified ASTM C 1567 Tests with 15%, 25% and 35% Fly Ash Dosage

Effectiveness of Fly Ash in Reducing ASR Expansions of Mortar Bars Containing New Mexico Rhyolite

Figure 4.23 shows the percent increase/decrease in the 14 day expansions of the mortar bars containing New Mexico rhyolite aggregate with the three types of fly ashes at 15%, 25% and 35% dosage each. The expansions are compared with the New Mexico control (0% fly ash) mortar bar expansion which is bench marked as 0%. A negative percent value indicates a reduction in the expansion and hence being effective.

It is clearly evident from the bar graphs that intermediate lime and low lime fly ashes are effective in reducing the expansions by almost 100% at 35% dosage in 1N NaOH. However, in potassium acetate exposure only low lime fly ash at 25% and 35% dosage is effective in reducing the expansions by almost 100% of control. High lime fly ash appears effective in reducing the expansions by a maximum of 73% at 35% dosage in 1N NaOH, but it appears to aggravate the expansions in potassium acetate exposure with 25% dosage having the highest increase (35%) in expansions.

Influence of ASTM C 1567 Test regime on Dynamic Modulus of Elasticity (DME)

Figure 4.24 shows the changes in the dynamic modulus of elasticity of mortar containing New Mexico rhyolite aggregate and three fly ashes of varied lime contents at 25% dosage in the Standard (1N NaOH) and modified (potassium acetate) ASTM C 1567 Tests.

Based on the results shown in figure 4.24 it is evident that an increase in the mortar bar expansion results in a simultaneous drop in the DME of the mortar bars. The sudden jump in the expansions of the mortar bars with intermediate and high lime fly ashes at 7 and 14 days in potassium acetate solution is noticeable in the DME results too. There is a pronounced drop in the DME at the same ages of 7 and 14 days for the mortar bars containing intermediate and low lime fly ash respectively. Likewise, the high lime fly ash mortar bars had very high expansions from 3 day test age itself and this is reciprocated by a steep drop in the DME values from 0 to 3 days.

Similar trends are observed for mortar bars soaked in 1N NaOH solution where the gradual increase in the expansions of mortar bars with these fly ashes is reciprocated by a gradual drop in the DME results.

Comparing the change in DME of mortar bars in 1N NaOH and potassium acetate exposure, the low lime fly ash mortar bars performed better in potassium acetate up to 14 days beyond which the increased expansions led to a loss of physical integrity and hence a drop in the DME .

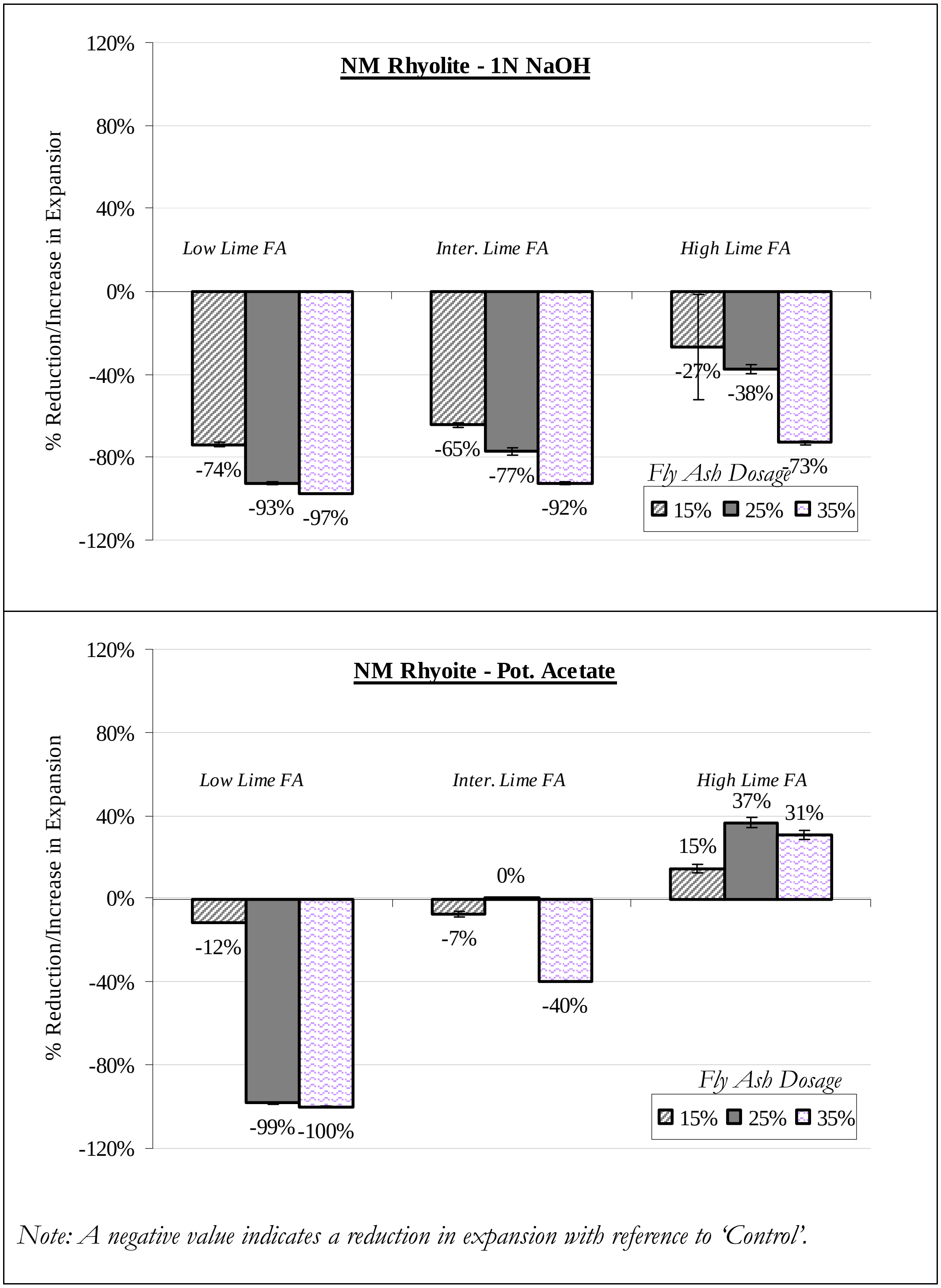



Figure 4.23 Percent Reduction/Increase in the 14 Day Mortar bar Expansions as a Factor of Fly Ash Type and Dosage in 1N NaOH and KAc Exposure with New Mexico Rhyolite as Aggregate.

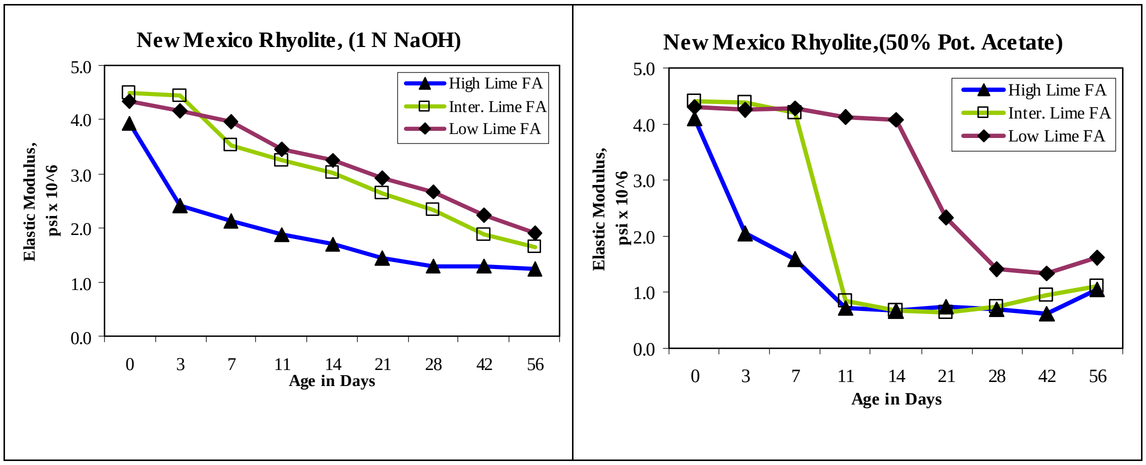



Figure 4.24 Changes in Dynamic Modulus of Elasticity of New Mexico Rhyolite Mortar Bars in Standard and Modified ASTM C 1567 Tests with 25% Fly Ash Dosage

### 4.3.4 North Carolina Argillite

Figure 4.25 shows the expansions of mortar bars with North Carolina argillite aggregate in 1N NaOH and potassium acetate solutions with each of the three fly ashes at 15%, 25% and 35% dosages respectively. The results are compared with the control mortar bar tests to provide a reference for the influence of fly ashes on the expansions.

Results shown in figure 4.25 indicate that high lime fly ash was not effective in reducing the expansions at 15% and 25% dosages in both 1N NaOH and potassium acetate exposure. In fact, the expansions of mortar bars at 25% dosage of high lime fly ash were higher than those of the control mortar bars. This behavior was similar to that of mortar bars with NM rhyolite aggregate and high lime fly ash. However, high lime fly ash was effective in reducing the expansions to below 0.1% at 35% dosage in both the solutions at 14 days and up to 28 days in potassium acetate exposure only. This indicates that the high lime fly ash is more effective in reducing the expansions potassium acetate exposure in comparison with 1N NaOH exposure.

Intermediate and low lime fly ashes were effective at 25% and 35% dosage in both 1N NaOH and potassium acetate solutions. However, intermediate lime fly ash was ineffective at 15% for both the soak solutions. In case of low lime fly ash, 15% dosage was not sufficient to mitigate the expansions in 1N NaOH but was sufficient to reduce the expansions in potassium acetate exposure.

Expansion results for all the fly ashes indicate that for cement-aggregate combinations of NC argillite aggregate and , low lime and intermediate lime fly ashes at 25% and 35% cement replacement levels can be effectively used to reduce the mortar bar expansions to below 0.1% in both 1N NaOH and potassium acetate solutions.

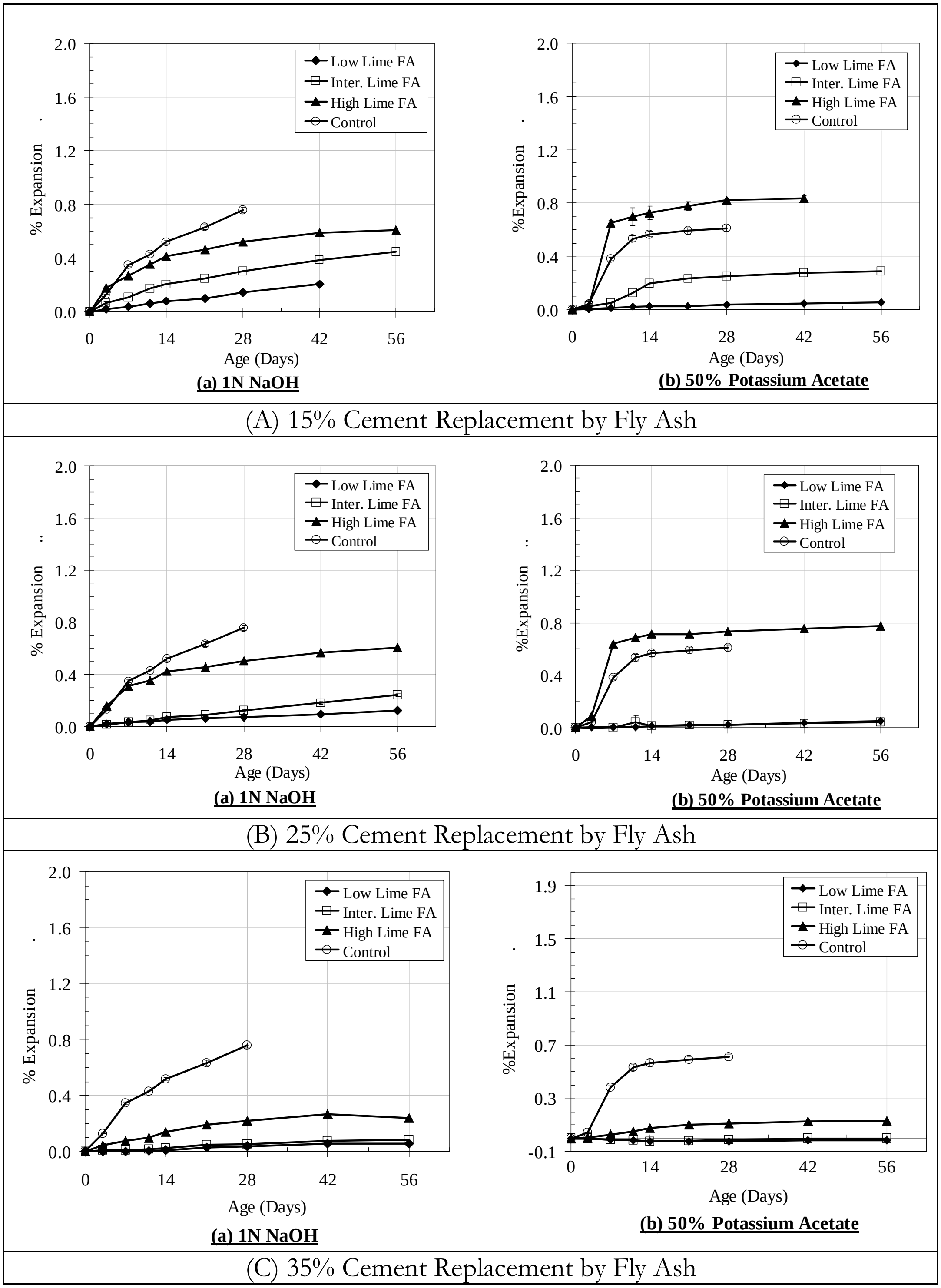



Figure 4.25 Expansions of Mortar Bars Containing North Carolina Argillite Aggregate in Standard and Modified ASTM C 1567 Tests with 15%, 25% and 35% Fly Ash Dosage

Effectiveness of Fly Ash in Reducing ASR Expansions of Mortar Bars Containing North Carolina Argillite

Figure 4.26 shows the percent increase/decrease in the 14 day expansions of the mortar bars containing North Carolina argillite aggregate with the three types of fly ashes at 15%, 25% and 35% dosage each. The expansions are compared with the NC control (0% fly ash) mortar bar expansion which is bench marked as 0%.

The expansion reduction trend of NC argillite is similar to that seen in NM rhyolite mortar bars (figure 4.24) where low and intermediate lime fly ashes provide a high reduction in expansions while high lime fly ash was effective only in 1N NaOH at 35% dosage. The effectiveness of low lime fly ash in 1N NaOH is almost similar at 25% and 35% dosage, while in potassium acetate all the three dosages are equally effective (95% to 105% reduction in expansions). High lime fly ash at 15% and 25% dosage appear to aggravate the expansions by increasing them more than the control. However, at 35% dosage high lime fly ash does become effective in reducing the expansions by 73% in both 1N NaOH and potassium acetate.

Influence of ASTM C 1567 Test regime on Dynamic Modulus of Elasticity (DME)

Results of changes in the DME of mortar bars in the standard and modified ASTM C 1567 test are shown in figure 4.27. The inverse relationship between the percent expansions of the mortar bars and its respective DME seems to prevail for this aggregate too. In 1N NaOH, the drop in DME is gradual for mortar bars with all the three fly ashes with high lime fly ash mortar bars showing the most drop. Similarly, for mortar bars in potassium acetate solution, those with the high lime fly ash had the most drop in DME followed by mortar bars with intermediate lime fly ash and low lime fly ash. A sudden drop in the DME of mortar bars with high lime fly ash and soaked in potassium acetate at 3 day test age is observed which is proportional to the sudden increase in the percent expansions in length change at the same test age.

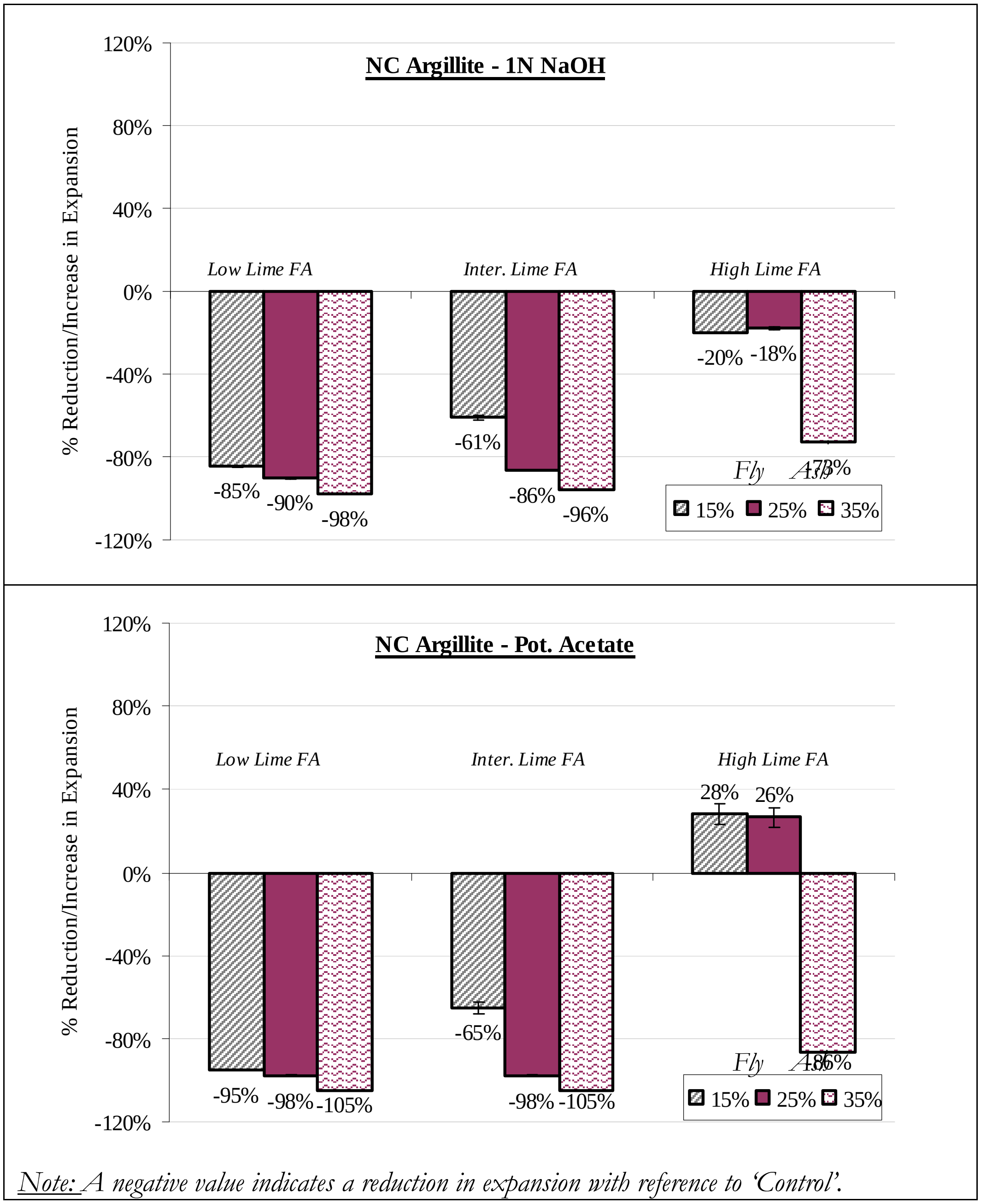



Figure 4.26 Percent Reduction/Increase in the 14 Day Mortar bar Expansions as a Factor of Fly Ash Type and Dosage in 1N NaOH and KAC Exposure with North Carolina Argillite as Aggregate.

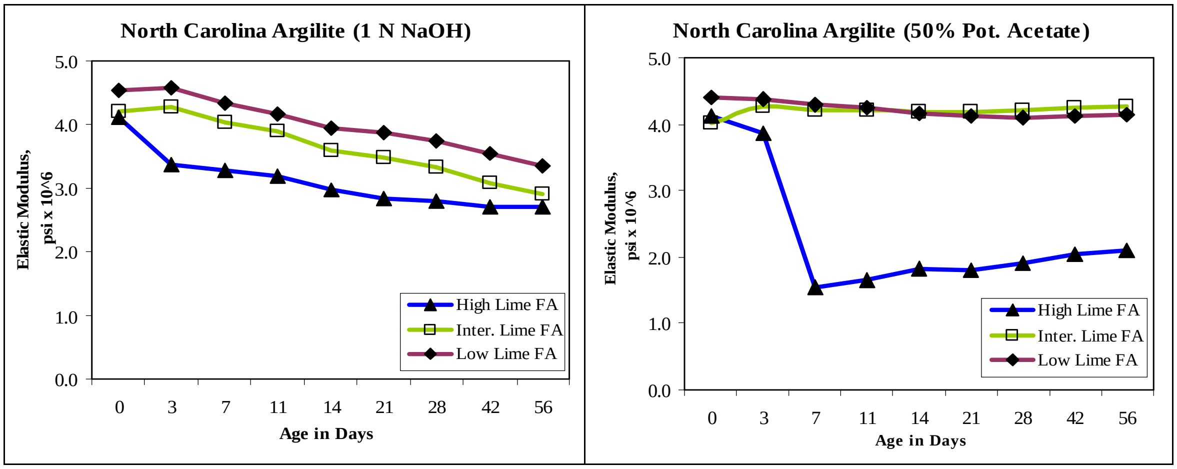



Figure 4.27 Changes in Dynamic Modulus of Elasticity of North Carolina Argillite Aggregate Mortar Bars in Standard and Modified ASTM C 1567 Tests with 25% Fly Ash Dosage

### 4.3.5 South Dakota Quartzite

Figure 4.28 shows the expansion results of the mortar bars made with South Dakota quartzite in the standard and modified ASTM C 1567 tests.

Results indicate that among the three fly ashes, low lime and intermediate lime fly ash at 25% and 35% dosage were effective in containing the expansions of the mortar bars to below 0.1% at 14 and 28 days test age in both the standard and modified ASTM C 1567 test. In the presence of potassium acetate solution, low lime fly ash at a dosage of 15% was effective in reducing the mortar bar expansions to 0.1% but this dosage was insufficient to contain the mortar bar expansions when exposed to 1N NaOH. Intermediate lime fly ash was not effective in reducing the expansions in both 1N NaOH and potassium acetate solutions at any of the three dosages.

High fly ash was ineffective in mitigating the mortar bar expansions at all the three cement replacement levels. The expansions observed for high lime fly ash mortar bars were higher when exposed to potassium acetate deicer than with 1N NaOH. At 15% and 25% fly ash dosage, the high lime fly ash containing mortar bars had expansions higher than the control mortar bars (with no fly ash) in potassium acetate solution. This trend of expansions with South Dakota aggregate and high lime fly ash is in agreement with that observed with mortar bars containing NM rhyolite and NC argillite aggregate.

It should be noted that the mortar bars containing SD quartzite aggregate with all the three fly ashes in potassium acetate deicer solution reached their ultimate expansions within a finite time and thereafter showed no further expansions. This behavior was in contrast to that observed in 1N NaOH solution where the mortar bar expansions increased gradually with time. Similar behavior was observed for the expansions of mortar bars with NM rhyolite and NC argillite aggregates.

### Effectiveness of Fly Ash in Reducing ASR Expansions of Mortar Bars Containing South Dakota Quartzite

Figure 4.29 shows the percent increase/decrease in the 14 day expansions of the mortar bars containing South Dakota quartzite aggregate with the three types of fly ashes at 15%, 25% and 35% dosage each. The expansions are compared with the SD control (0% fly ash) mortar bar expansion which is bench marked as 0%.

The expansion reduction behavior of SD quartzite containing mortar bars is similar to that of NC argillite (refer figure 4.26). In both the aggregates, low lime fly ash and intermediate lime fly ash are more effective in reducing the expansions in potassium acetate exposure than in 1N NaOH. Similarly, high lime fly ash at 15% and 25% dosage in potassium acetate increases the expansions while at the same dosage in 1N NaOH it has a minimal influence in reducing the expansions.

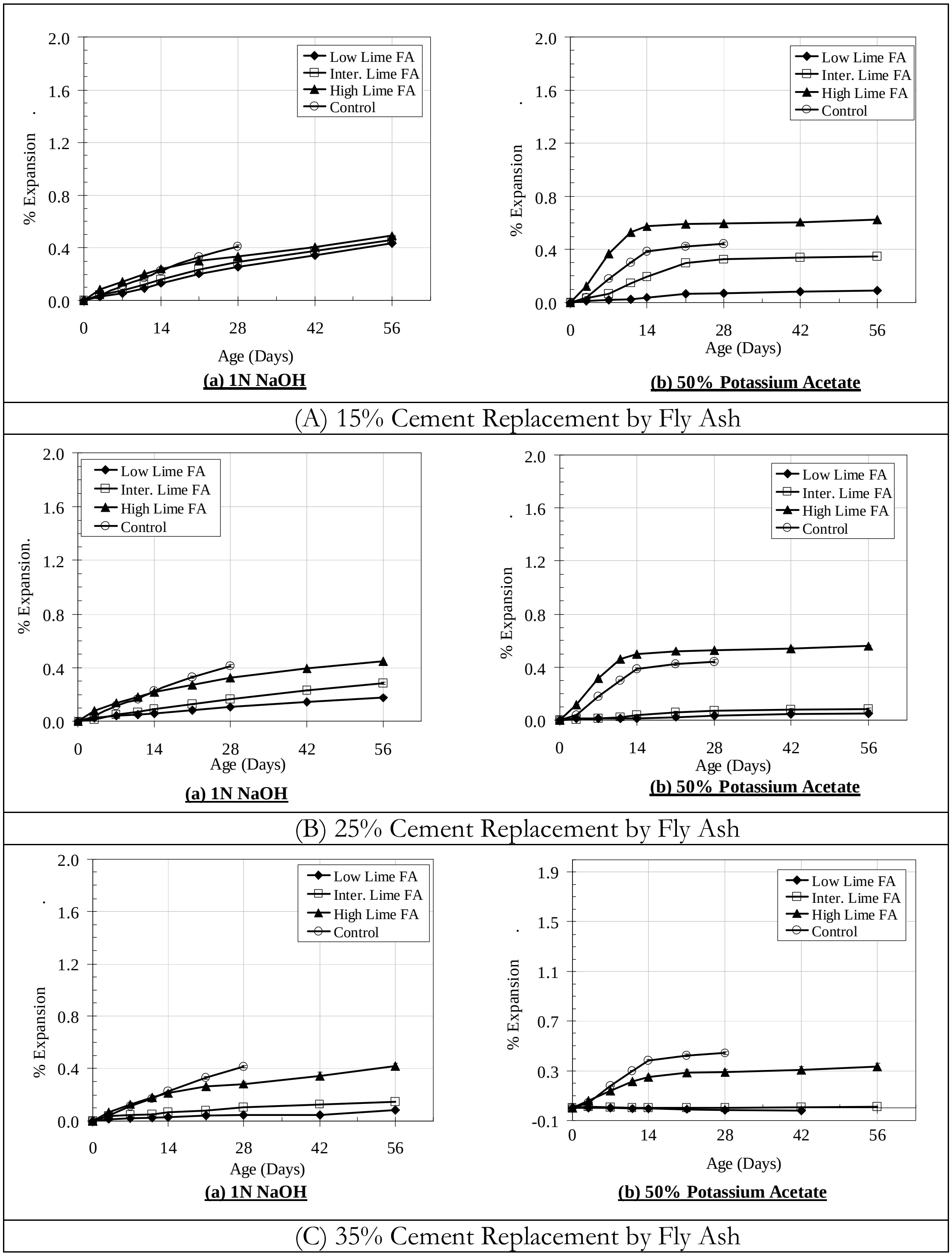



Figure 4.28 Expansions of Mortar Bars Containing South Dakota Quartzite Aggregate in Standard and Modified ASTM C 1567 Tests with 15%, 25% and 35% Fly Ash Dosage

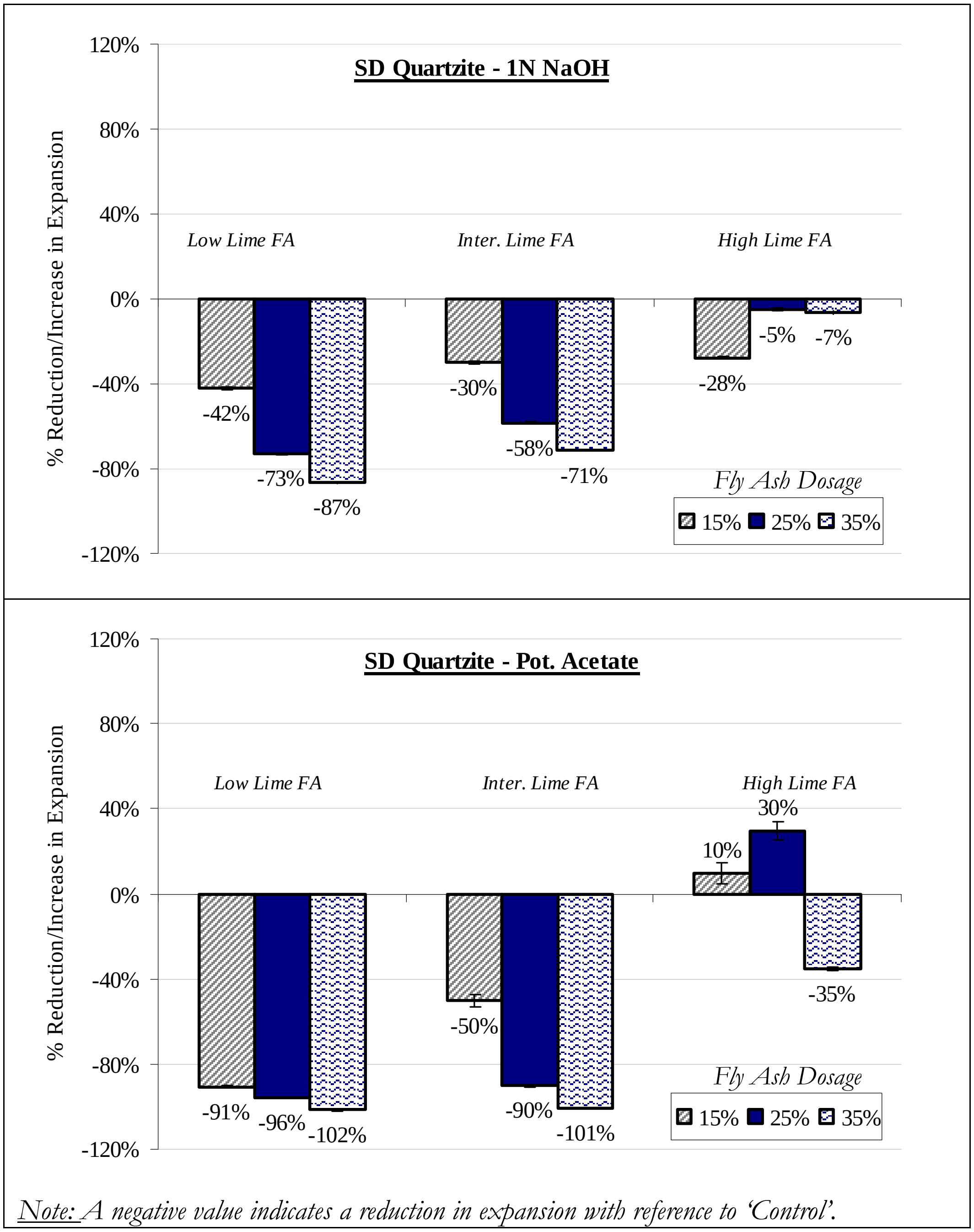



Figure 4.29 Percent Reduction/Increase in the 14 Day Mortar bar Expansions as a Factor of Fly Ash Type and Dosage in 1N NaOH and KAC Exposure with South Dakota Quartzite as Aggregate.

Influence of ASTM C 1567 Test regime on Dynamic Modulus of Elasticity (DME)

Figure 4.30 shows the change in DME of the mortar bars containing South Dakota aggregate and three fly ashes. The changes observed in the DME of the mortar with all the three fly ashes at 25% dosage showed similar behavior to that observed in the previously discussed three aggregates- SP limestone, NM rhyolite and NC argillite for both the solutions. An increase in the expansions is characterized by a similar decrease in the DME of the mortar bars.

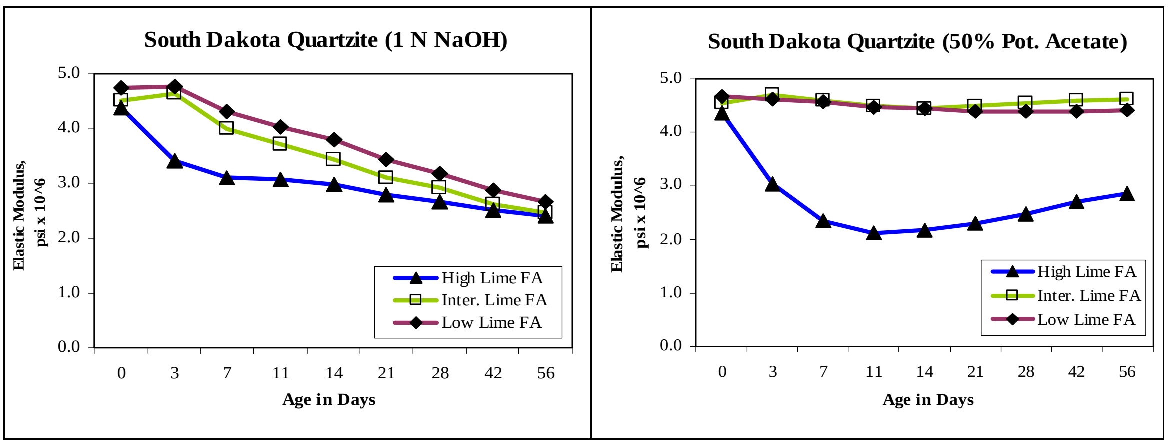



Figure 4.30 Changes in Dynamic Modulus of Elasticity (DME) of South Dakota Aggregate Mortar Bars in Standard and Modified ASTM C 1567 Tests with 25% Fly Ash Dosage

## 4.4 Results of Standard and Modified ASTM C 1567 Tests to Investigate the Effectiveness of Slag

This section presents the results from the standard and modified ASTM C 1567 tests for mortar bars containing slag at 40% and 50% cement replacement levels in combination with each of the four reactive aggregates- Spratt Limestone, New Mexico Rhyolite, North Carolina argillite and South Dakota Quartzite. To notice the influence of slag on reducing the mortar bar expansions, the expansions of the cement-slag and aggregate mixtures are compared with those of control mortar bars containing no slag. These tests were conducted beyond 28 days and up to 56 days to explore the potential for deleterious expansions at later ages.

### 4.4.1 Spratt Limestone

Figure 4.31 shows the expansions of mortar bars containing Spratt limestone aggregate and slag at 40% and 50% dosage in 1N NaOH and potassium acetate deicer solution. These are compared with the control (no slag) expansion results.

Results of the standard ASTM C 1567 tests indicate that slag at 40% and 50% dosage are able to significantly reduce, by 64% and 73% respectively, the mortar bar expansions to below 0.1% at 14 days in comparison to the control expansions. However, the expansions keep gradually increasing beyond 14 days indicating that slag is not effective in controlling the expansions in 1N NaOH exposure at later ages. In fact, the expansions with both 40% and 50% slag dosage showed a continued tendency to expand even at 56 days.

Results of the modified (potassium acetate) ASTM C 1567 tests indicate that slag significantly reduces the mortar bar expansions when compared to the control expansions.

At 40% slag dosage the expansion of mortar bars was slightly above the 0.1% mark (0.12%), but the expansions had an increasing trend similar to that observed in 1N NaOH tests. At a 50% dosage though the expansions had an increasing trend, the expansions (0.09%) were below the 0.1% limit up to 28 days beyond which they crossed the limit marginally (0.16%) at 56 days. The effectiveness of slag in mitigating the expansions was more pronounced in the case of potassium acetate exposure than for 1N NaOH exposure.

In sum, for Spratt limestone aggregate, a minimum dosage of 50% slag was required to mitigate the expansions in presence of potassium acetate.

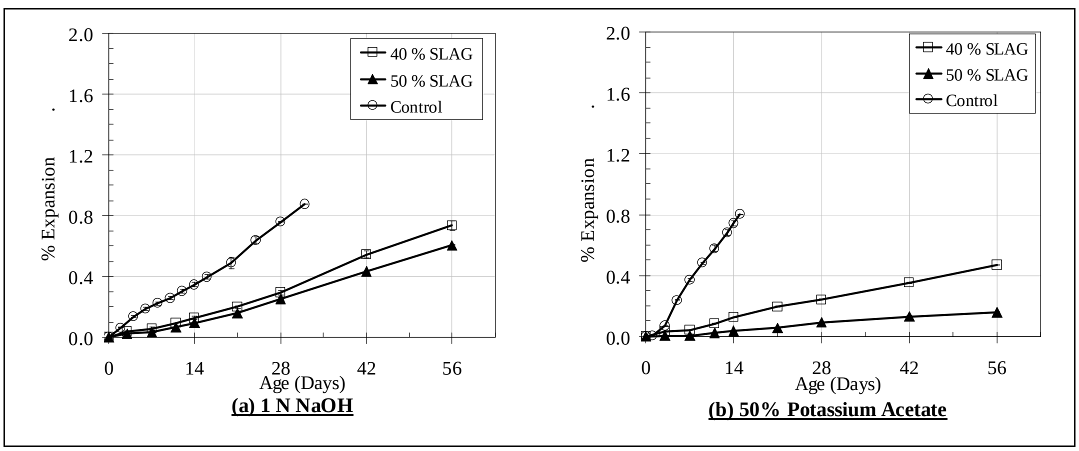



Figure 4.31 Expansions of Mortar Bars Containing Spratt Limestone Aggregate in Standard and Modified ASTM C 1567 Tests with Slag at 40% and 50% Dosage.

4.4.2 New Mexico Rhyolite

Figure 4.32 shows the length change behavior, in terms of percent expansion, of the mortar bars with New Mexico aggregate in combination with slag at 40% and 50% dosage.

Results of the standard ASTM C 1567 tests in the presence of 1NaOH solution indicate that slag was effective in mitigating the expansions with reference to the control mortar bars without any slag. However, slag was not effective enough to reduce the expansions to below 0.1% at 14 or 28 days at either of the cement replacement levels (40% and 50%). The increasing trend of expansions as observed in mortar bars with Spratt aggregate was evident for mortar bars with New Mexico aggregate too.

Results of the modified ASTM C 1567 tests in the presence of potassium acetate indicate that 40% slag offered a marginal mitigation compared to the control mortar bar and the expansion trend was similar to that of control. However, the expansion trend formed a plateau after reaching its maximum expansion at 14 days. Slag at 50% dosage was effective in mitigating the expansions with expansions below the 0.1% limit even at 56 days.

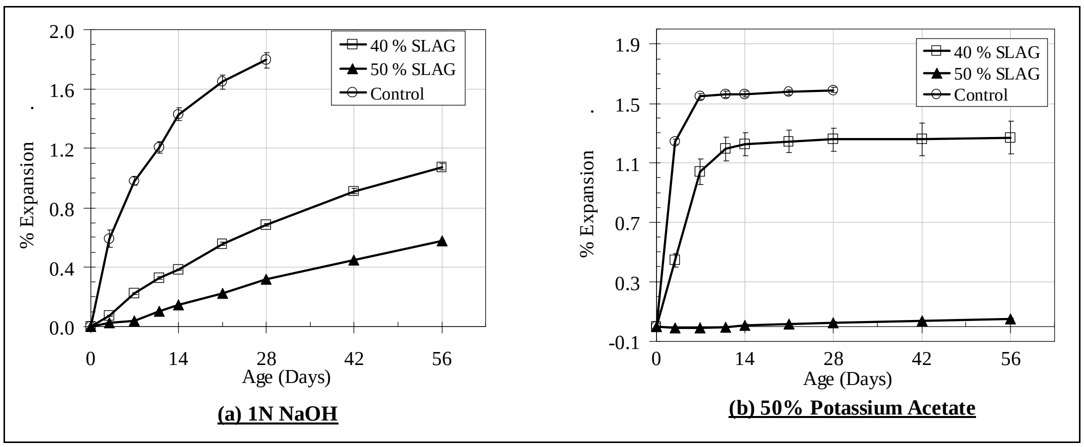



Figure 4.32 Expansions of Mortar Bars Containing New Mexico Rhyolite Aggregate in Standard and Modified ASTM C 1567 Tests with Slag at 40% and 50% Dosage.

### 4.4.3 North Carolina Argillite

Figure 4.33 shows the expansions of mortar bars with North Carolina argillite aggregate in combination with slag at 40% and 50% cement replacement levels. These are compared with the control mortar bar expansions to provide a reference.

Slag at 40% and 50% dosage was found to be effective in reducing the expansions in comparison to the control mortar bars in the standard ASTM C 1567 test. However, only 50% dosage of slag could mitigate the expansions to below 0.1% at 28 days.

In the case of mortar bars exposed to potassium acetate deicer in the modified ASTM C 1567 test, both 40% and 50% dosage were effective enough to contain the expansions to below 0.1% at 28 days with 50% dosage showing a higher mitigation compared to 40%.

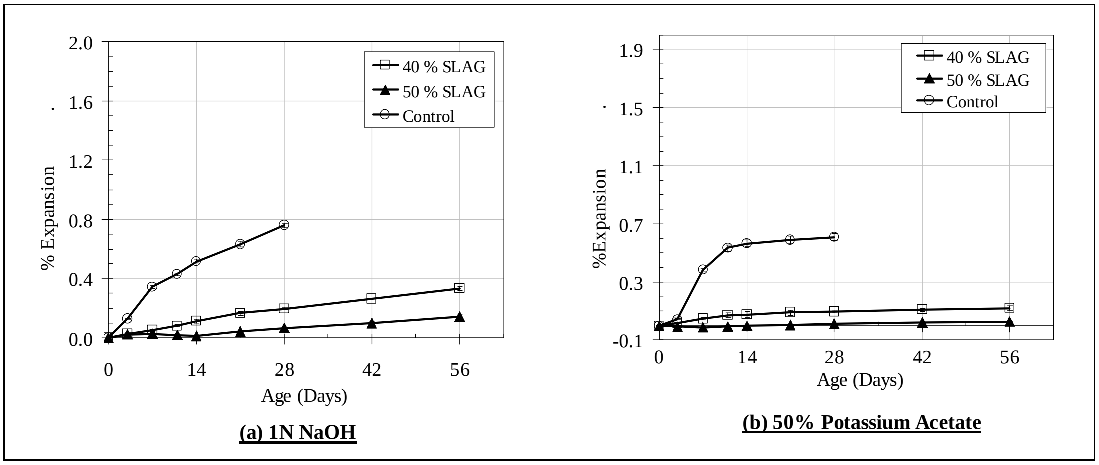



Figure 4.33 Expansions of Mortar Bars Containing North Carolina Argillite Aggregate in Standard and Modified ASTM C 1567 Tests with Slag at 40% and 50% Dosage.

## 4.4.4 South Dakota Quartzite

Figure 4.34 show the expansion behavior of South Dakota quartzite containing mortar bars in combination with slag at 40% and 50% cement replacement level. The results are compared with the expansions of the control mortar bars containing no slag.

Standard ASTM C 1567 tests in the presence of 1N NaOH concluded that though slag was effective in reducing the expansions of mortar bars in comparison to the control, it was not effective in reducing the expansions to below 0.1% at 28 days at both the cement replacement levels. The increasing trend of mortar bar expansions that was observed in the

1N NaOH tests with Spratt and New Mexico aggregate was also evident for mortar bars with South Dakota aggregate.

Results of the modified ASTM C 1567 test indicate that 40% slag was not just ineffective, but also detrimental to the mitigation of mortar bars. The ultimate expansions of the mortar bars with 40% slag were higher than the control. A 50% dosage was adequate to mitigate the expansions to below 0.1% at 14 and 28 days in the presence of KAc deicer.

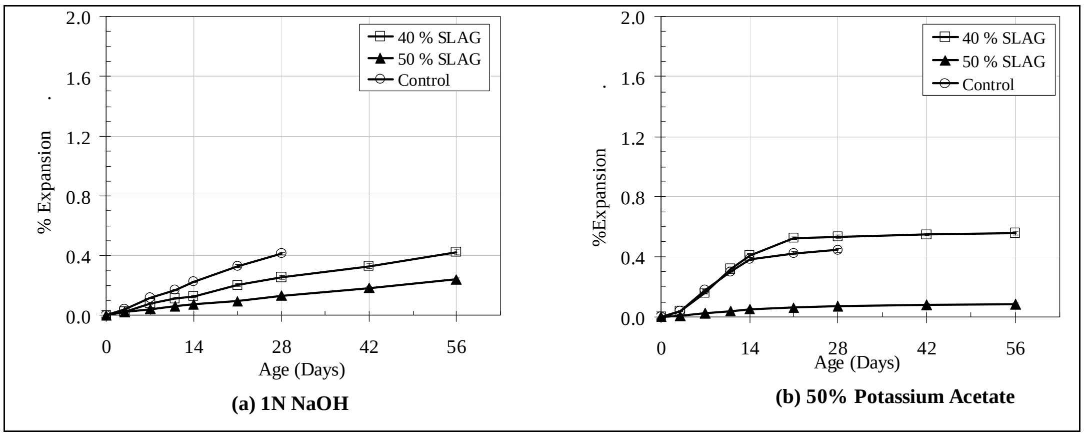



Figure 4.34 Expansions of Mortar Bars Containing South Dakota Quartzite Aggregate in Standard and Modified ASTM C 1567 Tests with Slag at 40% and 50% Dosage.

<u>Effectiveness of Slag on Mortar Bar Expansions</u>

Figure 4.35 shows the reduction/increase in the 14 day expansions of mortar bars with SP, NM, NC and SD aggregate and slag at 40% and 50% dosage in 1N NaOH and potassium acetate. The percent reduction in expansions is with reference to the respective control mortar bar expansions.

Irrespective of the exposure condition, slag appears to reduce the mortar bar expansions for all the four aggregates at 50% dosage. In 1N NaOH, slag reduces the expansions at both 40% and 50% dosage for all the four aggregates. However, in potassium acetate, except for SD quartzite at 40% dosage, slag is effective in reducing the expansions of all the four aggregates at both the dosages. SD quartzite appears to have a negative influence on the mortar bar expansions at 40% dosage as it increases the expansions higher than the control.

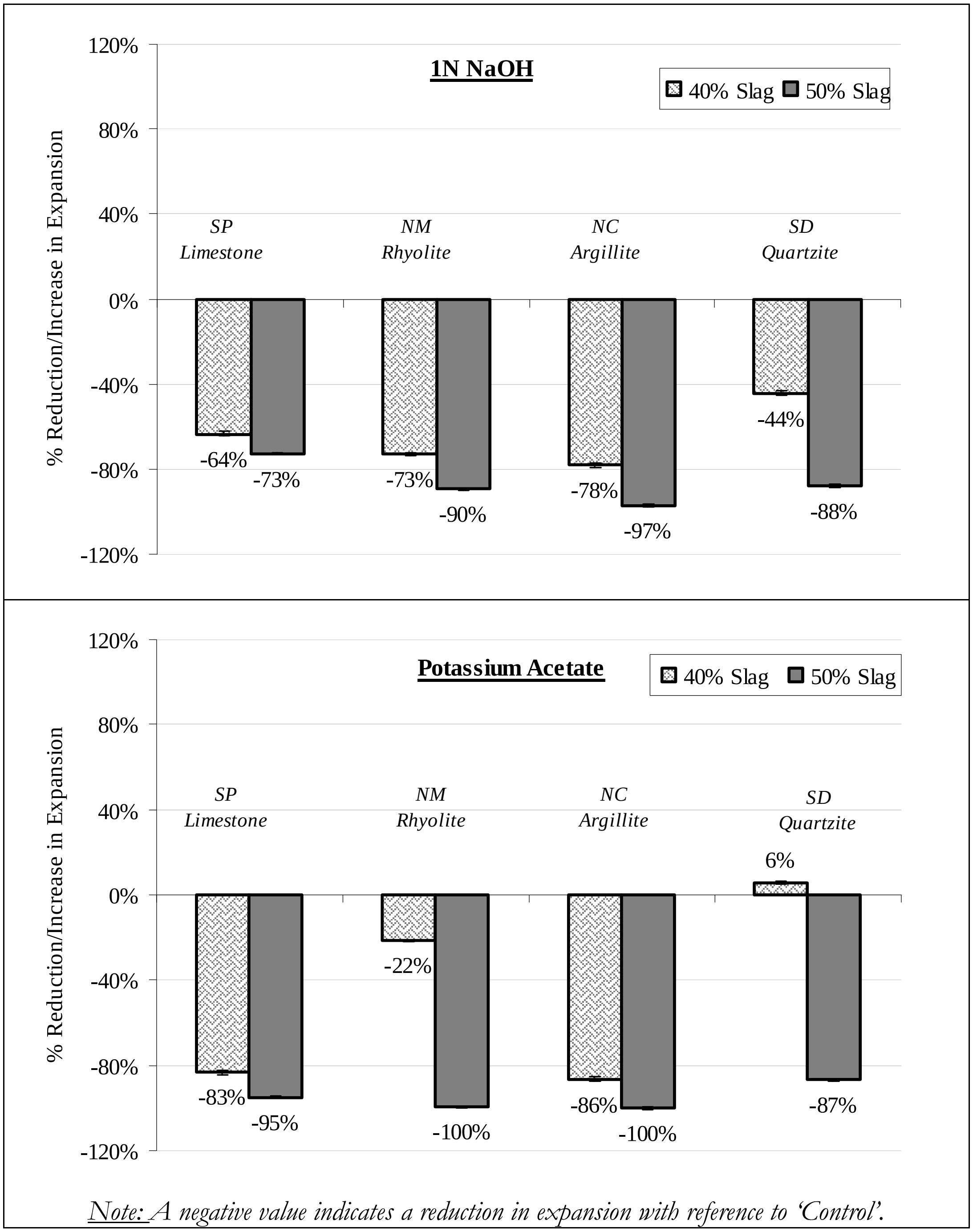



Figure 4.35 Percent Reduction/Increase in the 14 Day Mortar bar Expansions as a Factor of Aggregate Type and Slag Dosage in 1N NaOH and KAC Exposure with Spratt Limestone as Aggregate.

## 4.5 Results Modified ASTM C 1293 Tests to Investigate the Effectiveness of Fly Ashes

This section presents the results of the modified ASTM C 1293 tests on concrete prisms containing either of the two reactive aggregates- Spratt limestone and New Mexico Rhyolite, in combination with three fly ashes- low lime, intermediate and high lime fly ash; at 25% ad 35% cement replacement.

The modified tests are of two types as described in section 3.3.6 and 3.3.7 and they differ from each other with respect to the solutions in which the concrete prisms are soaked. Modified (Type 1) ASTM C 1293 test has 1N NaOH as soak solution while modified (Type 2) ASTM C 1293 has potassium acetate deicer as soak solution. However, for the simplicity of discussion these tests will be addressed to as 1N NaOH and potassium acetate exposure tests respectively. The length change results of these are compared with the control concrete prisms containing no fly ash to provide a reference for the mitigation potential fly ashes. The control test results of NM Rhyolite and SP Limestone in these tests are from a doctoral research conducted at Clemson University (Sompura 2006).

Results presented for each of the two aggregates include the following:

- Length change behavior of the concrete prisms in 1N NaOH and potassium acetate deicer solutions.
- Dynamic modulus of elasticity (DME) of concrete prisms with three fly ashes at 25% and 35% cement replacement in 1N NaOH and potassium acetate solutions and,
- Microstructure studies of Spratt limestone containing concrete prisms with fly ashes at 25% cement replacement level.

The expansion limit for the concrete prisms containing SCMs in the standard ASTM C 1293 test is 0.04% at 2 years. The same limit is used for the modified ASTM C 1293 tests in the presence of 1N NaOH and potassium acetate deicer solution.

### 4.5.1 Spratt Limestone

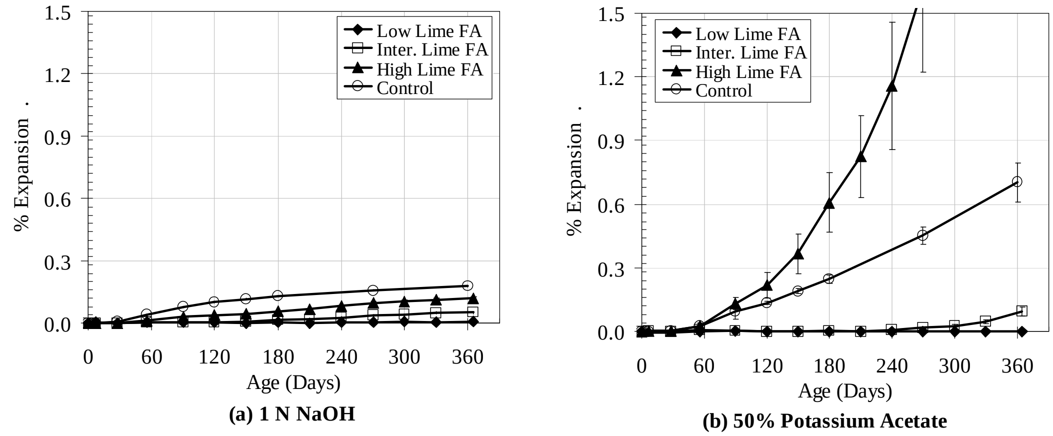
Figure 4.36 and Figure 4.37 show the expansion behavior of concrete prisms containing Spratt limestone aggregate in combination of three fly ashes at 25% and 35% cement replacement level in 1N NaOH and potassium acetate solution, respectively

Results of the expansions of concrete prisms soaked in 1N NaOH indicate that all the three fly ashes were effective in reducing the expansion of the concrete prisms in comparison to the control expansions. The mitigation behavior of fly ashes at 25% and 35% dosage was identical. Intermediate lime fly ash at 25% dosage was inadequate to control the expansions to below 0.04% (0.05%) at 1 year test age. However, at 35% fly ash dosage intermediate lime fly ash mitigated the expansions to below 0.04% (0.03%) at 1year. Low lime fly ash was highly effective in mitigating the expansions of concrete prisms at both 25% (0.008%) and 35% (0.003%) dosage at 1 year test age. High lime fly ash was effective in reducing the expansions to less than the control prisms at both 25% and 35% dosage, but was not adequate to control the expansions to below 0.04%.

Expansions of the concrete prisms in potassium acetate deicer exposure indicate that low lime and intermediate lime fly ash at 25% and 35% dosage were effective in reducing the expansions to less than the control prisms. Intermediate lime fly ash at 25% dosage had expansions identical to that of low lime fly ash prisms up to 300 days, beyond which an increase was observed that crossed the 0.04% limit. This behavior indicates the potential for expansion at a later age when intermediate lime fly ash at 25% dosage is used in the presence of potassium acetate. However, at 35% dosage of intermediate lime fly ash the expansions were identical to that of low lime fly ash prisms at the same dosage and were below 0.04% at 1 year test age.

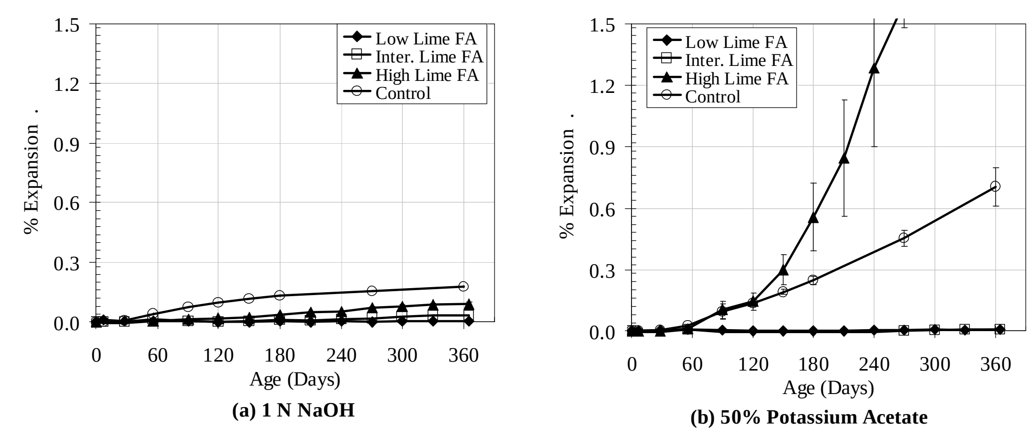
High lime fly ash appears to aggravate the expansions in the presence of potassium acetate and the expansion trend of the concrete prisms was in contrast to its positive mitigation behavior in 1N NaOH exposure. Unlike the concrete prisms with low and intermediate lime fly ash, the concrete prisms containing high lime fly ash had high expansions and were characterized by extensive cracking and subsequent loss of physical integrity as shown in the following DME results. The expansions were so high that the concrete prisms broke into half at 330 days test age. This behavior was observed at both 25% and 35% fly ash dosage. In fact, the expansions increased with an increase in the fly ash dosage. This indicates a strong negative influence of the high lime fly ash in the presence of potassium acetate deicer.

It should be noted that due to the excessive expansion of one of the concrete prism, among the set of four prisms, containing 'High Lime' fly ash at 25% and 35% dosage at 210 days, the variability of the average expansion result was high.




*Note: Excessive expansion observed in one of the four prisms of 'High Lime' fly ash in potassium acetate at 210 days and beyond leading to variability in test results.*

Figure 4.36 Expansions of Concrete Prisms Containing Spratt Limestone Aggregate in Modified ASTM C 1293 Tests with Fly Ashes at 25% Dosage.




*Note: Excessive expansion observed in one of the four prisms of 'High Lime' fly ash in potassium acetate at 210 days and beyond leading to variability in test results.*

Figure 4.37 Expansions of Concrete Prisms Containing Spratt Limestone Aggregate in Modified ASTM C 1293 Tests with Fly Ashes at 35% Dosage.

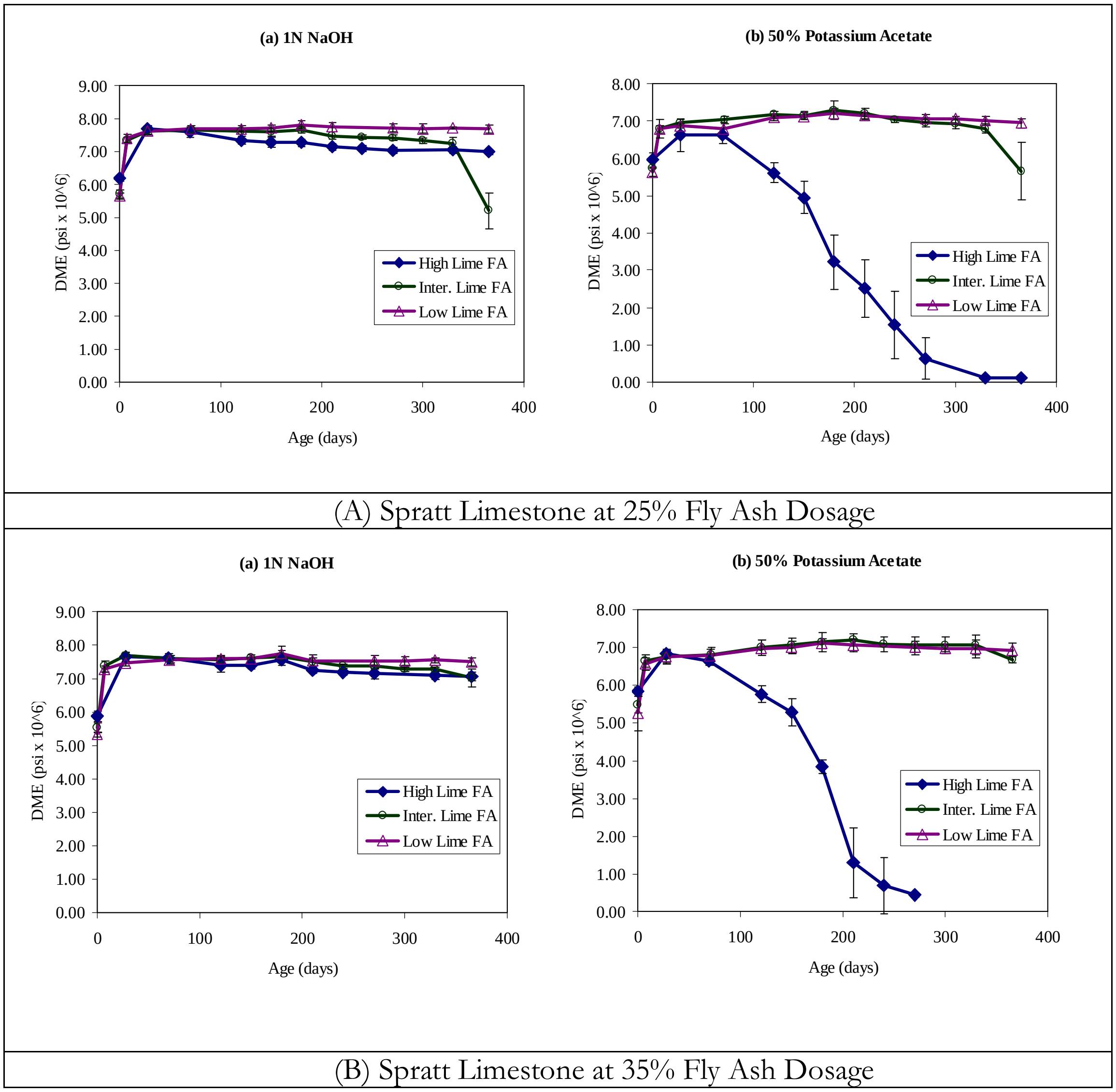
## Influence of Modified ASTM C 1293 Tests on the DME

Figure 4.38 (A) and 4.38 (B) show the changes in the DME of the concrete prisms containing Spratt aggregate in combination with three fly ashes at 25% and 35% cement replacement levels respectively. DME is measured on the same ages as the length change measurement.

Based on the results of the DME in 1N NaOH at 25% and 35% fly ash dosage, it can be concluded that the drop in the DME is for all the three fly ashes is marginal. This is in agreement with the low expansions observed in the modified ASTM C 1293 test.

Contrary to what is observed for the concrete prisms in 1N NaOH exposure, the changes in the DME of the concrete prisms in potassium acetate are much more evident. The DME of low and intermediate lime fly ashes were almost identical at both the fly ash dosages. An increase in the expansion of the concrete prism is characterized by a drop in the DME. For instance, an increase in expansion of intermediate lime fly ash containing concrete prism in potassium acetate at 330 days is reciprocated by a drop in the DME at the same age (see figure 4.37 and 4.38(A)).Similarly, the high expansions of the high lime fly ash containing concrete prisms are marked by a steep drop in the DME.

It should be noted that the onset of cracking in the concrete prisms is characterized by a drop in the DME. However, when the cracking of the concrete prisms becomes extensive, the measurement of frequency generated by impulse excitation method becomes difficult and hence variable. It is due to this reason that once the concrete has cracked severely, the variability in the DME results increases.




(A) Spratt Limestone at 25% Fly Ash Dosage

(B) Spratt Limestone at 35% Fly Ash Dosage

Figure 4.38 Changes in DME of Concrete Prisms Containing Spratt Limestone Aggregate in Modified ASTM C 1293 Tests with Fly Ashes at 25% and 35% Dosage

4.5.2 Microstructure Studies- Modified ASTM C 1293 Tests Involving Spratt Limestone with Fly Ash

This section presents the results of the microstructure studies conducted on the concrete prism samples from the modified ASTM C 1293 tests. The samples studied include sections sliced from concrete prisms soaked in 1N NaOH and KAc for 365 days with Spratt limestone as aggregate and fly ash at 25% cement replacement. Samples were selected to represent a low lime, intermediate and high lime fly ash in combination with Spratt limestone aggregate at 25% dosage.

Spratt Limestone Concrete Prisms Containing High Lime Fly Ash

Figure 4.39 and 4.40 shows the visual images of the concrete prisms containing Spratt limestone aggregate with high lime fly ash and exposed to 1N NaOH and potassium acetate respectively. This is followed by SEM micrographs and EDX spectra of the samples from the concrete prisms.

*1N NaOH Soak Solution*

It is evident from the visual images shown in figure 4.39 that the concrete prisms containing high lime fly ash at 25% dosage did not show any signs of physical distress on its surface. Minor hair cracks were faintly visible on the edge faces of the prisms. This evidence supports the low expansions recorded in the modified ASTM C 1293 test.

Figure4.41 shows the SEM micrographs and EDX spectra of some regions of the concrete sample. It is evident from the SEM micrographs that though there are negligible signs of physical damage on the surface of the concrete prisms, signs of cracking are visible in the mortar matrix as well as within the aggregate particles. Figure 4.41 (A) shows the formation of ASR reaction product on the periphery of the aggregate particle. An EDX spectrum (EDX2) on the formation shows a silica rich phase similar to that of cement paste. It is suspected that the reaction product might be formed due to the dissolution of silica of the aggregate particle and the reaction mechanism might be restricted to the surface only.

*Potassium Acetate (KAc) Soak Solution*

Visual images of the concrete prisms shown in figure 4.40 clearly indicate the extensive damage caused to the concrete on exposure to potassium acetate deicer solution. The extensive damage is marked by wide open cracks running through the concrete prisms and de-bonding of coarse aggregate particles and cement mortar.

SEM micrographs shown in figure 4.42 substantiate the evidence seen in the visual images and show the extensive cracking within the cement mortar matrix and wide cracks running through the aggregate into the cement matrix. EDX analyses conducted on the walls of the cracks formed within the aggregate particles did not reveal the presence of ASR gel. It is believed that the reaction product so formed would have diffused through the wide and extensive cracks within the sample. Figure 4.42 (A and A1) show a cracked aggregate particle and the paste surrounding it. An ASR like reaction product is seen adjacent to the aggregate in the cement paste and it shows high amount of potassium. EDX spectra of spots near the aggregate periphery in the cement mortar matrix show high amounts of potassium that is infused by the potassium acetate solution.


Figure 4.39 Visual Images of Concrete Prisms Containing Spratt Limestone Aggregate with 25% High Lime Fly Ash (HL3) Soaked in 1N NaOH for 365 Days


Figure 4.40 Visual Images of Concrete Prisms Containing Spratt Limestone Aggregate with 25% High Lime Fly Ash (HL3) Soaked in KAc for 365 Days

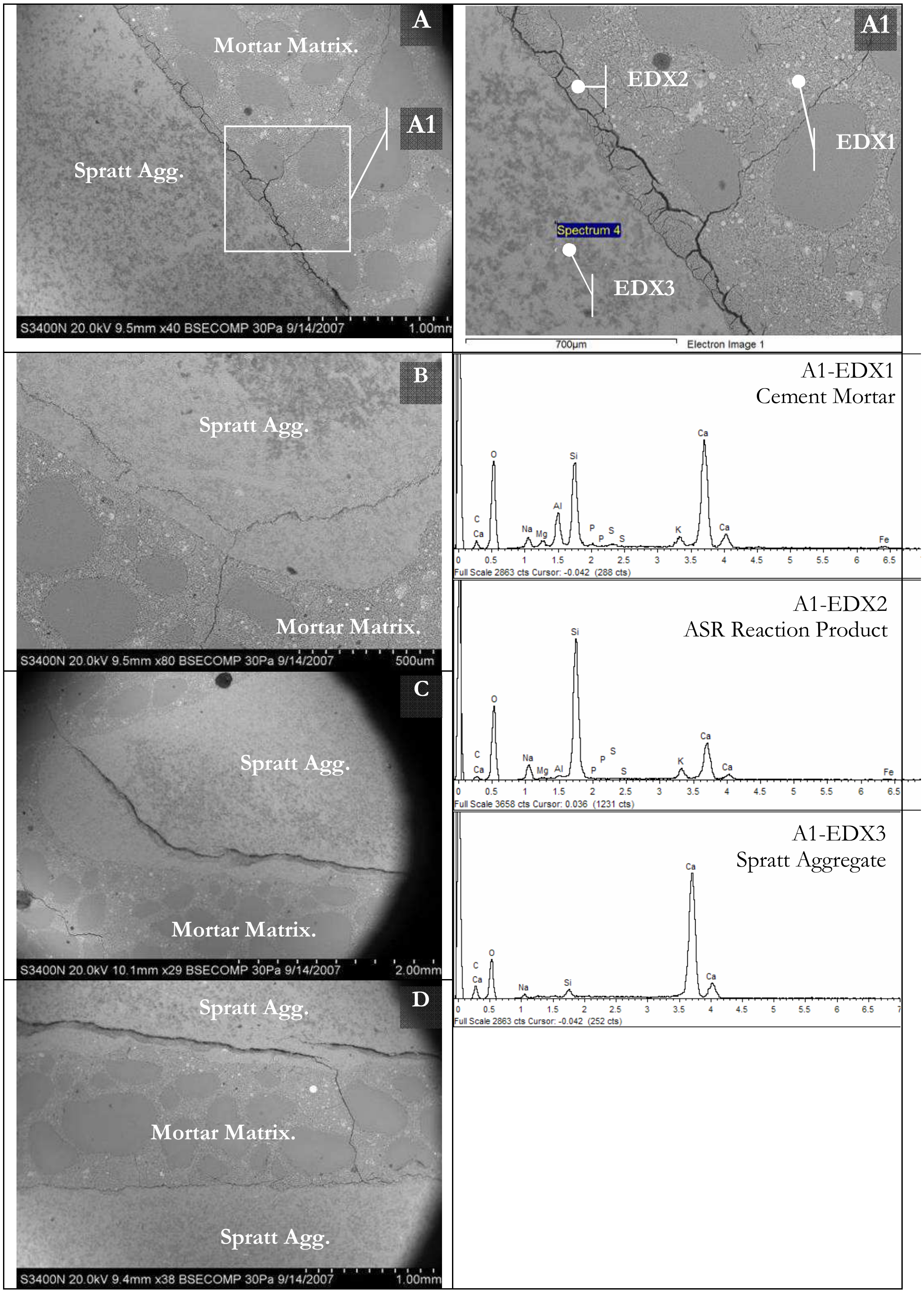



Figure 4.41 SEM Micrographs and EDX Analysis of Concrete Prism Samples Containing Spratt Limestone With 25% High Lime Fly Ash (HL3) in 1N NaOH (A) ASR Gel around the Aggregate (B), (C) and (D) Cracking through the Aggregate and Cement Matrix

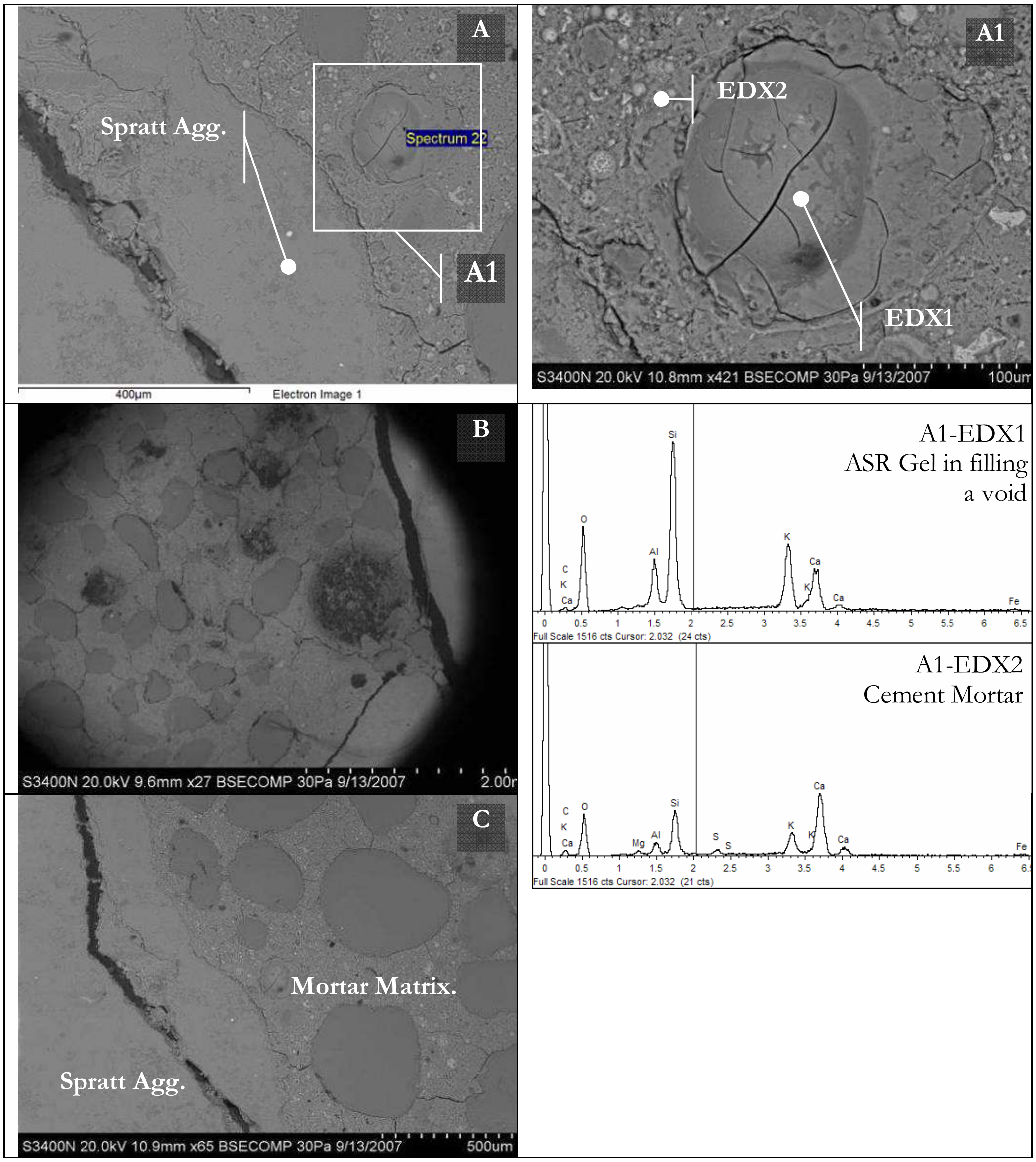



Figure 4.42 SEM Micrographs and EDX Analysis of Concrete Prism Samples Containing Spratt Limestone With 25% High Lime Fly Ash (HL3) in KAc
(A) Crack through Aggregate and Cracked Fly Ash Particle in Cement Matrix, (B) Severe Cracking Throughout the Cement Matrix and Around Ottawa Sand Grains, (C) Cracking through the Aggregate Leading into Cement Matrix

Spratt Limestone Concrete Prisms Containing Intermediate Lime Fly Ash

Figure 4.43 and 4.44 shows the visual images of the concrete prisms containing Spratt limestone aggregate with intermediate lime fly ash and exposed to 1N NaOH and potassium acetate respectively. This is followed by SEM micrographs and EDX spectra of the samples from the concrete prisms.

*1N NaOH Soak Solution*

Figure 4.43 shows the visual images of the concrete prisms soaked in 1N NaOH indicate no visible signs of physical distress. This supports the low expansions recorded in the modified ASTM C 1293 test. However, the SEM micrographs shown in figure 4.45 indicate a similar observation as seen in the 1N NaOH exposed concrete samples containing high lime fly ash where inspite of not having any visible signs of distress on the surface, the internal structure of the concrete reveal cracks in the mortar matrix and within the aggregate particles. Figures 4.45 (A) and (C) indicate the presence of an ASR gel within the crack running across the aggregate particle and in the crack originating from the aggregate into the cement paste, respectively.

*Potassium Acetate (KAc) Soak Solution*

The visual images showed in figure 4.44 indicate the presence of severe cracking on the surface of the concrete prisms. Though the density of the cracks in not high, the visible cracks are in are at the ends of the prisms and some cracks run across the length of the specimen.

Figure 4.46 shows SEM micrographs and EDX spectra of concrete samples exposed to potassium acetate and clearly indicate the distress in the form of cracking in the mortar matrix and within the aggregate particle. Figure 4.46 (A) clearly indicates the formation of an ASR gel at the aggregate-paste interface and at the end of the crack originating from within the aggregate. High amounts of potassium were detected at this cement aggregate interface and in the ASR gel. Figure 4.46 (C) indicates that the reaction product is collected at the aggregate-paste interface and is surrounding an Ottawa sand silica grain. EDX spectrum indicates a rich presence of silica in the reaction product. This observation is in noted for the high lime fly ash concrete samples too where a silica rich reaction product is detected at the aggregate-paste interface. This substantiates the theory that the interaction between KAc and aggregate leads to the dissolution of silica of the aggregate particles and diffuses into the cement matrix.

The distinguishing feature between the 1N NaOH and potassium acetate exposed concrete samples was the de-bonding of the aggregate particles and the cement paste. This de-bonding is characteristic of the potassium acetate distress and is believed to be occurring due to the expansion of the cement paste.

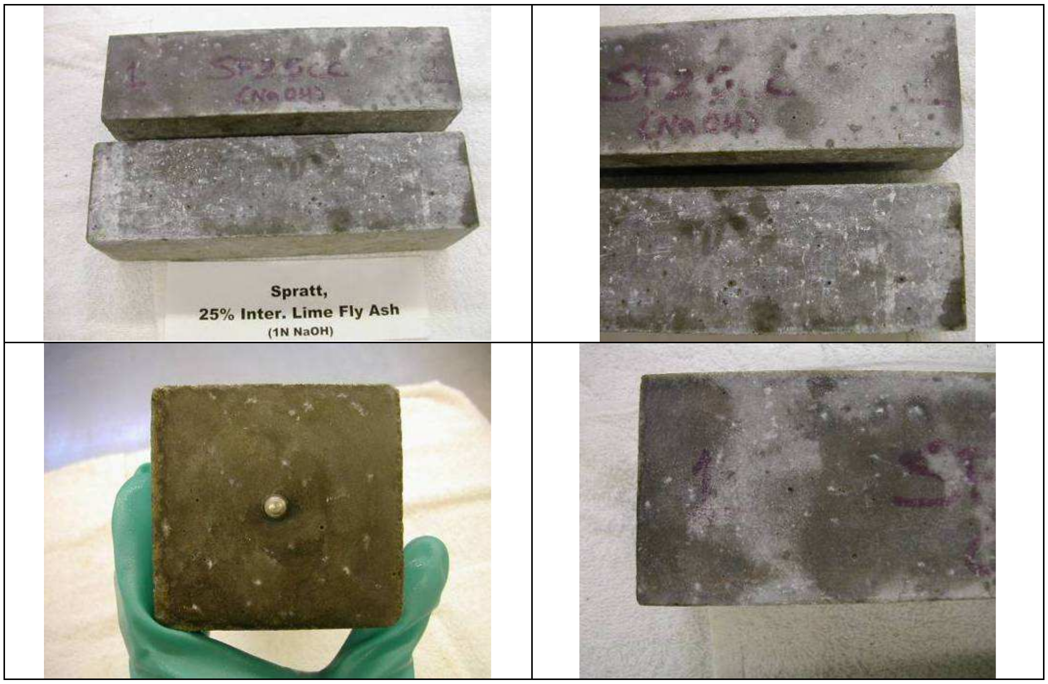



Figure 4.43 Visual Images of Concrete Prisms Containing Spratt Limestone Aggregate with 25% Intermediate Lime Fly Ash Soaked in 1N NaOH for 365 Days


Figure 4.44 Visual Images of Concrete Prisms Containing Spratt Limestone Aggregate with 25% Intermediate Lime Fly Ash Soaked in KAc for 365 Days

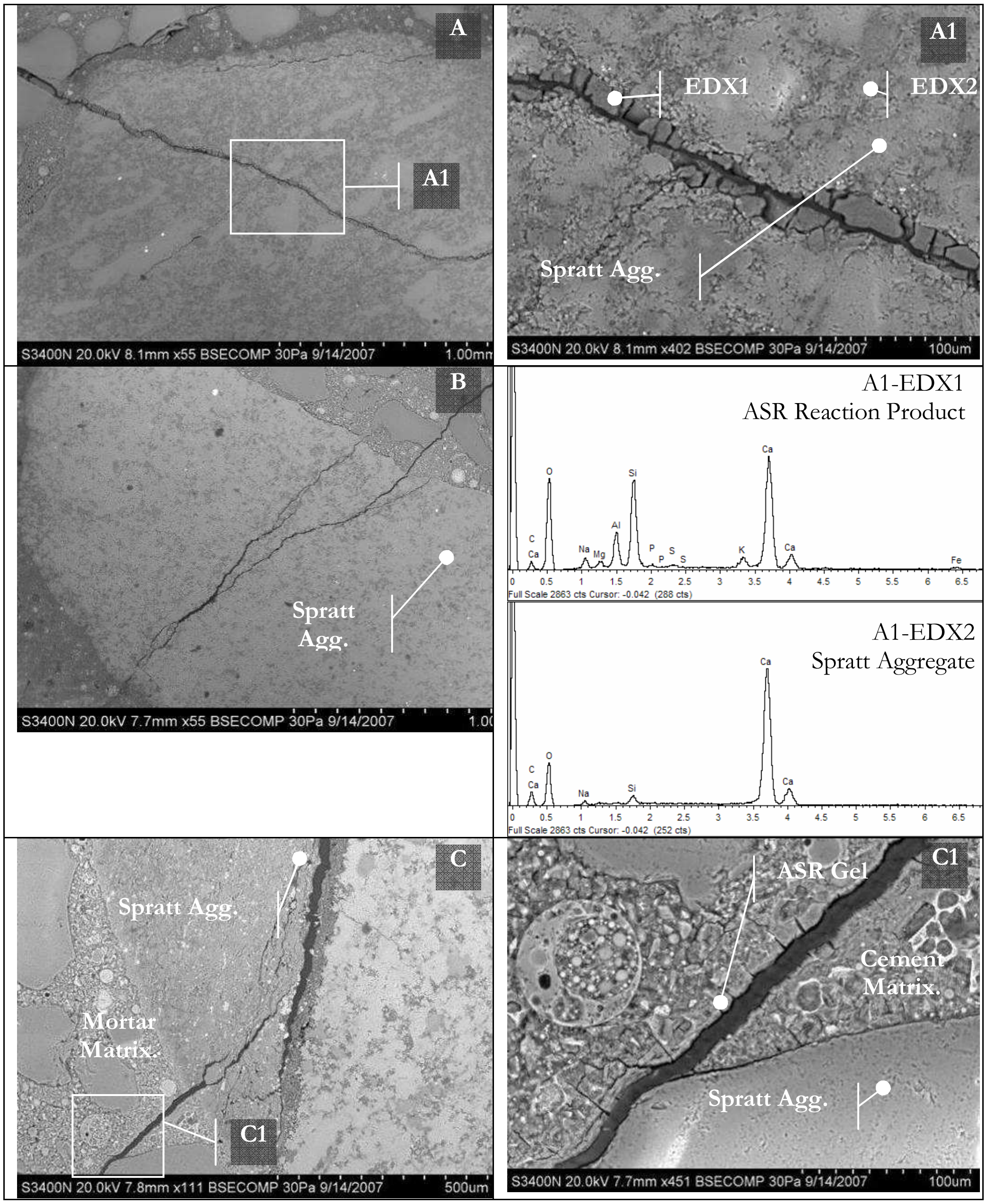



Figure 4.45 SEM Micrographs and EDX Analysis of Concrete Prism Samples Containing Spratt Limestone With 25% Intermediate Lime Fly Ash (IL5) in 1N NaOH

(A and A1) ASR Gel in the Crack through the Aggregate, (B) Cracking through the Aggregate into the Cement Matrix (C and C1) ASR Gel in the Crack through the Aggregate into the Cement Matrix.

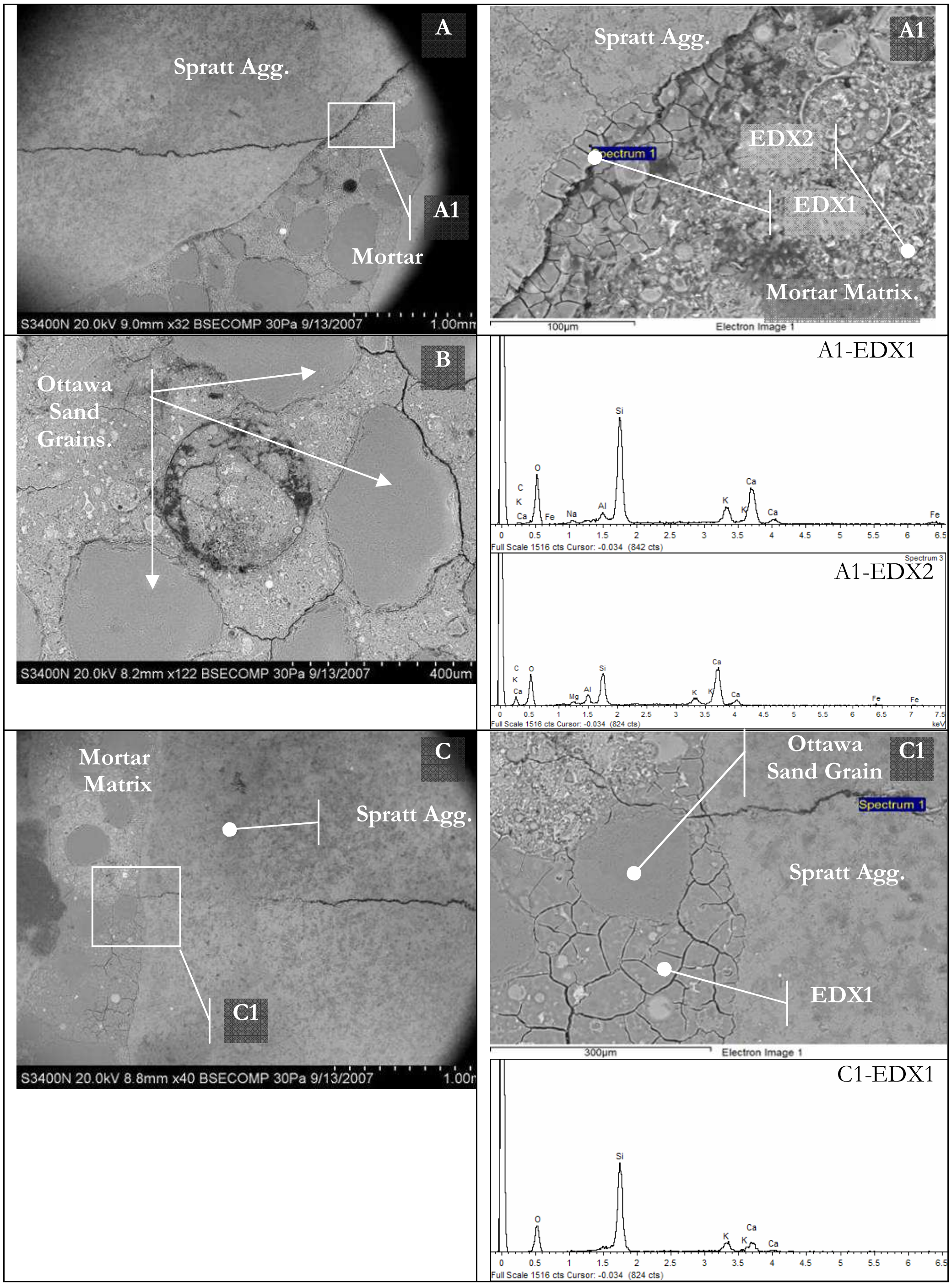



Figure 4.46 SEM Micrographs and EDX Analysis of Concrete Prism Samples Containing Spratt Limestone With 25% Intermediate Lime Fly Ash (IL5) in KAc
(A and A1) ASR Gel in the Crack and near Aggregate Paste Interface, (B) Cracking through out the Cement Matrix and De-bonding of Sand Grains (C and C1) Crack through the Aggregate into the Cement Matrix.

## Spratt Limestone Concrete Prisms Containing Low Lime Fly Ash

Figure 4.47 and 4.48 shows the visual images of the concrete prisms containing Spratt limestone aggregate with intermediate lime fly ash and exposed to 1N NaOH and potassium acetate respectively. This is followed by SEM micrographs of the samples from the concrete prisms.

### *1N NaOH Soak Solution*

Figure 4.47 shows the visual images of the concrete prisms soaked in 1N NaOH and they indicate no signs of physical distress on the surface. However, the internal structure of the concrete prisms as seen under the SEM indicates the formation of a dark reaction rim on the aggregate-paste interface (see figure 4.49A and B). The mortar matrix did not show any significant cracking through out the sample. However, minor cracks within the aggregate particles and leading into the cement paste were observed in some aggregate particles (see figure 4.49 (C and C1)).

### *Potassium Acetate (KAc) Soak Solution*

Figure 4.48 shows the visual images of the concrete prisms soaked in 1N NaOH and they indicate no signs of physical distress on the surface. Similar to the observation made in concrete sample exposed to 1N NaOH, concrete exposed to potassium acetate and containing low lime fly ash did not show any signs of significant cracking through out the mortar matrix besides few minor cracks. Figure 4.50 (C) shows a SEM micrograph of a typical undamaged mortar matrix observed in the concrete sample with low lime fly ash.

Figure 4.50(A) indicates that inspite of the sound mortar matrix with minimal cracking; the cement matrix is rich in potassium infused by the soak solution. A crack generated in an aggregate particle and leading into the cement paste shows signs of formation of ASR gel as detected in the EDX spectra (EDX2), but is not significant enough to cause detrimental expansions.


Figure 4.47 Visual Images of Concrete Prisms Containing Spratt Limestone Aggregate with 25% Low Lime Fly Ash Soaked in 1N NaOH for 365 Days

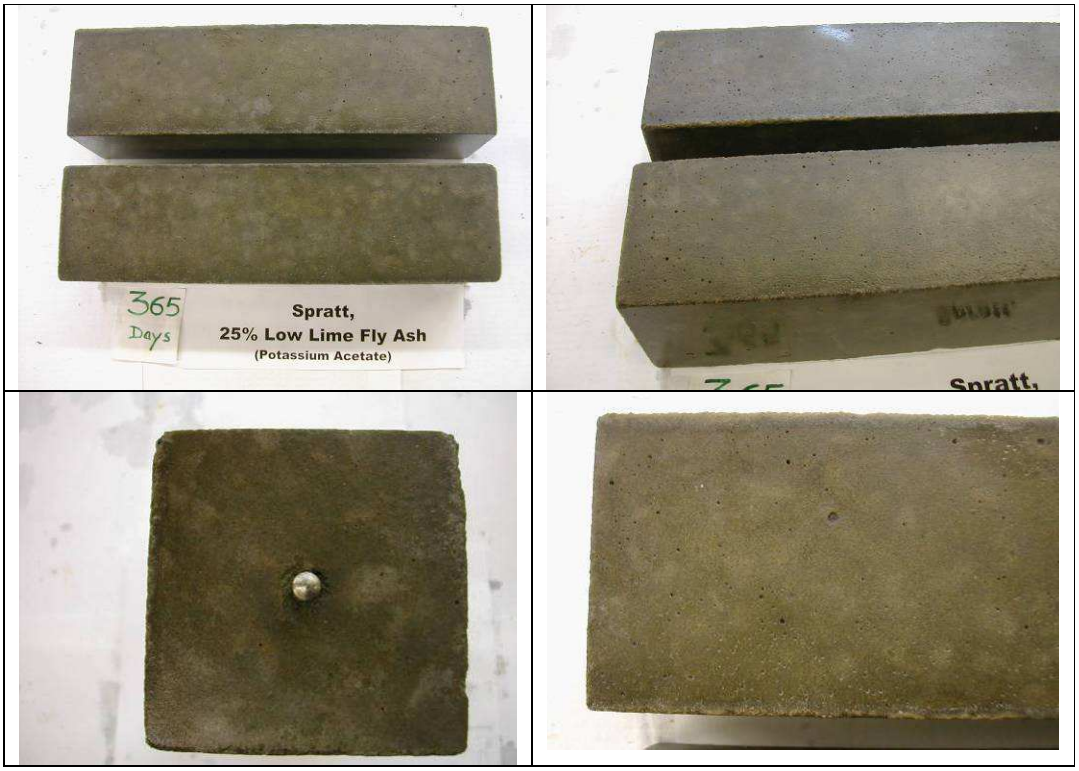



Figure 4.48 Visual Images of Concrete Prisms Containing Spratt Limestone Aggregate with 25% Low Lime Fly Ash Soaked in KAc for 365 Days

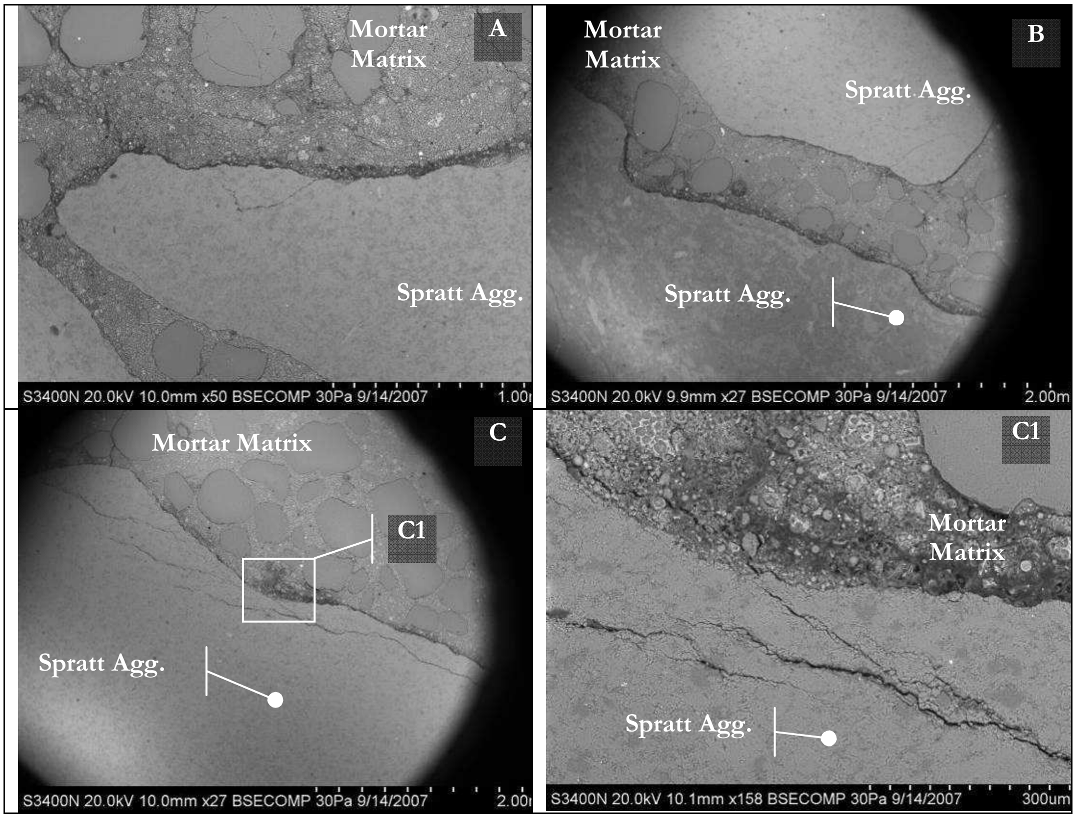



Figure 4.49 SEM Micrographs of Concrete Prism Samples Containing Spratt Limestone With 25% Low Lime Fly Ash (LL3) in 1N NaOH

(A) and (B) Darkened Reaction Rim Formed around the Aggregate Particle, (C and C1) Cracking of the Aggregate Particle and Dark Reaction Rim around the Aggregate.

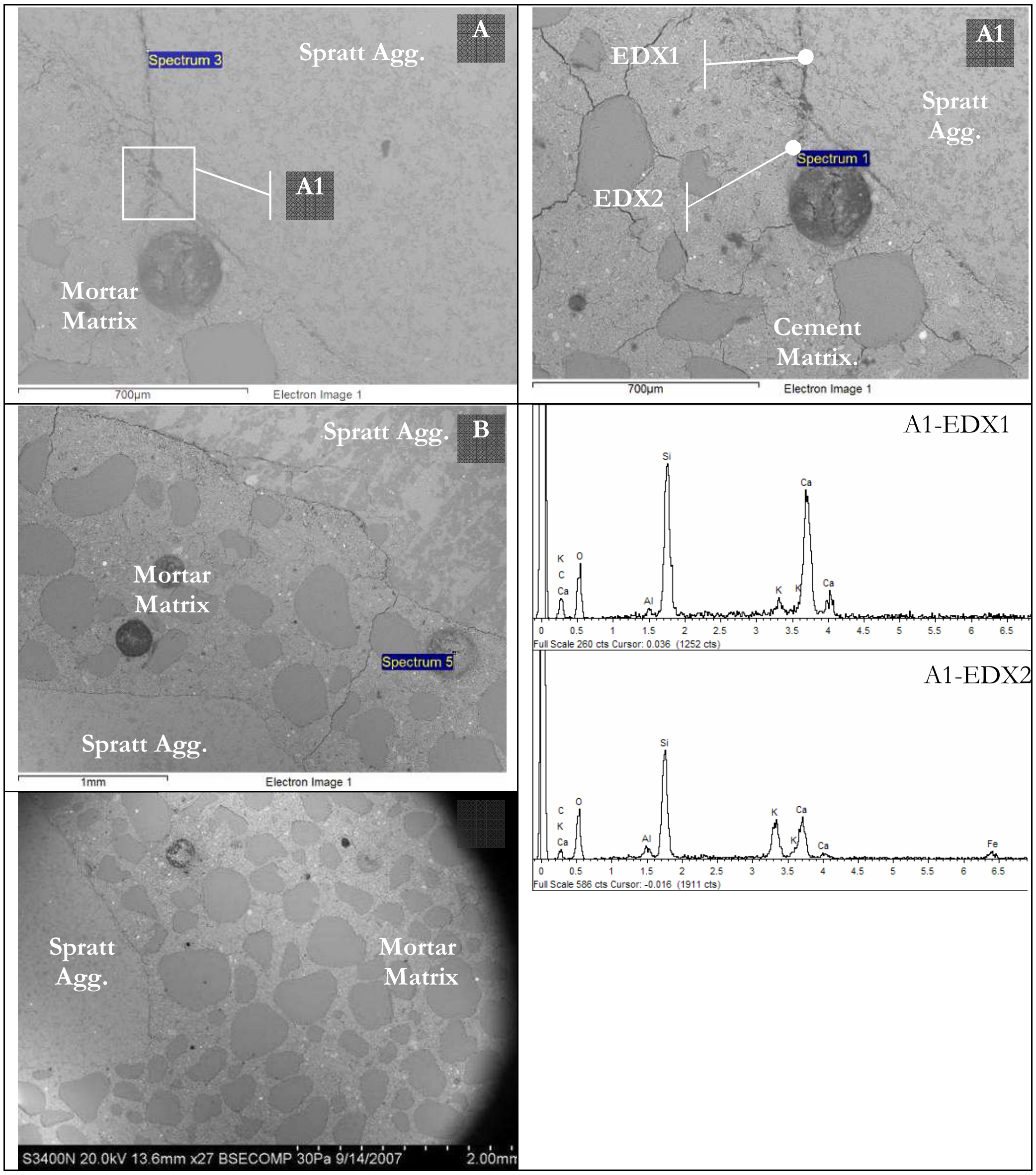



Figure 4.50 SEM Micrographs and EDX Analysis of Concrete Prism Sample Containing Spratt Limestone With 25% Low Lime Fly Ash (LL3) in KAc

(A and A1) ASR Gel Extruded through the Crack of the Aggregate Leading into the Cement Matrix (B) Minor Cracking of the Aggregate Particle and in the Cement Matrix, (C) No Signs of Cracking Seen in the Cement matrix.

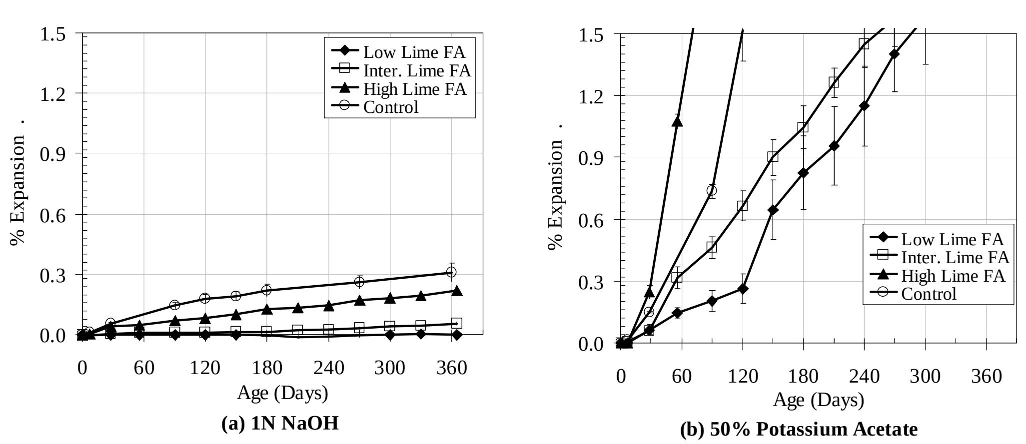
### 4.5.3 New Mexico Rhyolite

Figure 4.51 and 4.52 shows the expansions of the concrete prisms in the modified ASTM C 1293 tests containing New Mexico rhyolite aggregate in combination with three fly ashes at 25% and 35% cement replacement levels respectively.

Results indicate that all the three fly ashes were effective in reducing the expansions of the concrete prisms exposed to 1N NaOH in comparison to the control prisms containing no fly ash. This mitigation behavior was observed for both the fly ash dosages-25% and 35%. Low lime fly ash was effective at 25% and 35% dosages and the expansions of the concrete prisms containing this fly ash were below 0.04% at 1 year test age when exposed to 1N NaOH. Intermediate lime fly ash was effective to suppress the concrete prism expansions to below 0.04% at 35% fly ash dosage. However, at 25% dosage the expansions were marginally higher (0.05%) than the 0.04% limit at 1 year test age. Though high lime fly ash was effective in reducing the expansions of the concrete prisms with respect to the control prisms, it was ineffective in mitigating the expansions to below 0.04% at both 25% and 35% fly ash dosage.

The behavior of the concrete prisms containing three fly ashes at 25% and 35% dosage in the presence of potassium acetate deicer solution indicate that fly ashes were not effective in containing the deleterious ASR expansions. Low lime and intermediate lime fly ashes reduced the expansions of the concrete prisms to less than the control prisms, but were highly ineffective in limiting the expansions to 0.04%. Though the expansions of the concrete prisms containing both these fly ashes were high (1.94% at 1year for low lime fly ash at 25% dosage, 1.83% at 1 year for intermediate lime fly ash at 25% dosage), the expansions reduced with an increase in the fly ash dosage to 35% (1.22% at 1year for low lime fly ash at 35% dosage, 1.51% at 1 year for intermediate lime fly ash at 35% dosage). The ineffectiveness of the low lime fly ash as an ASR mitigation measure was contrary to what is published in the literature and to the results of the ASTM C 1567 mortar bar test. The damage to the concrete prisms containing low lime fly ash at 25% was such that it led to the breaking of the prisms at 330 days test age.

In the case of high lime fly ashes, the expansions of the concrete prisms were higher than the control and they increased with an increase in the fly ash dosage. This trend was similar to what was observed for concrete prisms containing Spratt limestone aggregate. The high expansions led to extensive cracking at very early ages of the test and were followed by breaking of the concrete prisms at 90 days and 120 days test age for 25% and 35% dosage respectively.




*Note: Excessive expansion observed in one of the four prisms of 'Low Lime' fly ash in potassium acetate at 150 days and beyond, leading to variability in test results.*

Figure 4.51 Expansions of Concrete Prisms Containing New Mexico Rhyolite Aggregate in Modified ASTM C 1293 Tests with Fly Ashes at 25% Dosage.

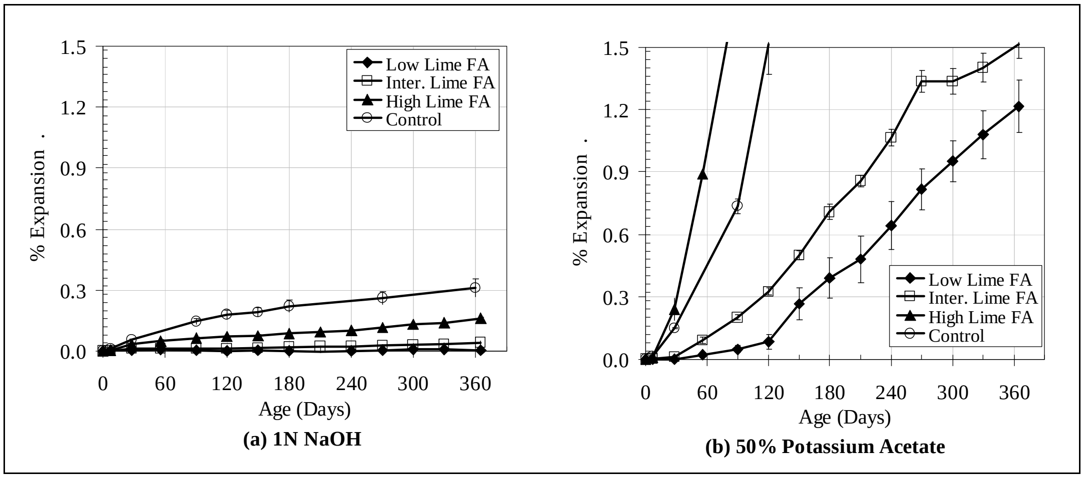



Figure 4.52 Expansions of Concrete Prisms Containing New Mexico Rhyolite Aggregate in Modified ASTM C 1293 Tests with Fly Ashes at 35% Dosage.

Influence of Modified ASTM C 1293 Tests on the DME

Figure 4.53 (A) and (B) show the changes in the DME of the concrete prisms containing three fly ashes at 25% and 35% cement replacement levels, respectively.

The changes in the DME of the concrete prisms exposed to 1N NaOH suggest that the drop or indifference in the DME results over the test age was indicative of the increase or stability of the expansions of the concrete prisms.

In the case of concrete prisms with all the three fly ashes at 25% and 35% dosage and exposed to potassium acetate solution, the steep drop in the DME is representative of the steep increase in the expansions in the modified ASTM C 1293 test. The drop in the DME is indicative of the loss of physical integrity of the concrete prisms over the testing period.

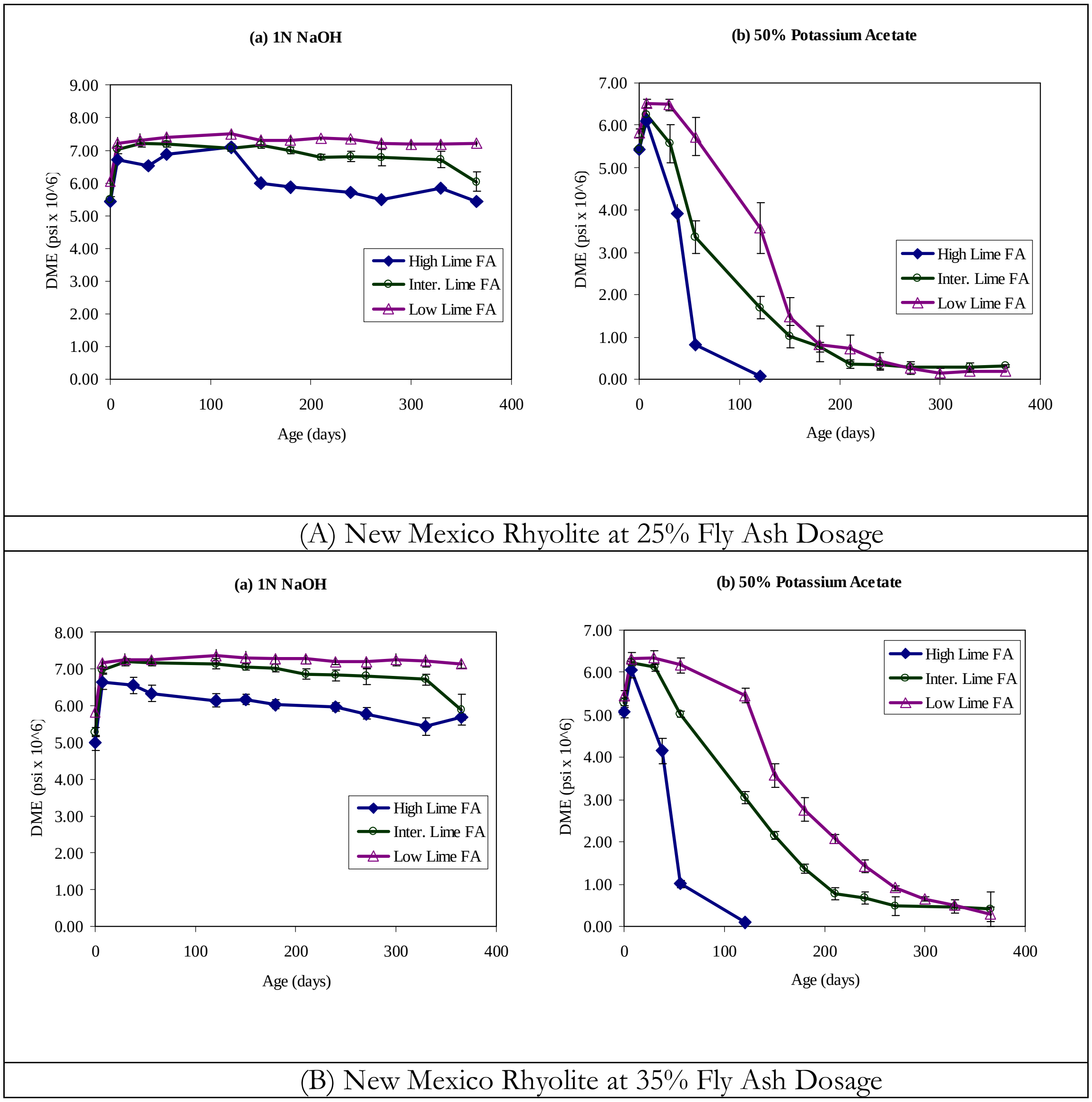



Figure 4.53 Changes in DME of Concrete Prisms Containing New Mexico Rhyolite Aggregate in Modified ASTM C 1293 Tests with Fly Ashes at 25% and 35% Dosage

## 4.6 Results Modified ASTM C 1293 Tests to Investigate the Effectiveness of Slag

This section presents the results of the modified ASTM C 1293 tests containing two reactive- Spratt Limestone and New Mexico rhyolite, and one non-reactive aggregate- Illinois dolomite; in combination with slag at 40% and 50% cement replacement level. The length change behavior of the concrete prisms during the modified ASTM C 1293 is presented along with the changes in the dynamic modulus of elasticity (DME) results.

### 4.6.1 Spratt Limestone

Figure 4.54 show the results of the concrete prisms containing Spratt limestone aggregate in combination with slag at 40% and 50% dosage in the presence of 1N NaOH and potassium acetate, respectively. The results are compared with the control concrete prisms containing no slag.

Results of the length change behavior of the concrete prisms in 1N NaOH indicate that both 40% and 50% slag is adequate to mitigate the expansions to below 0.04%. However, since the concrete prisms might expand over the remaining 9 months of the test, it might be misleading to conclude that 40% slag is effective in reducing the expansions.

Slag appears to be effective in reducing the expansions of the concrete prisms in the presence of potassium acetate deicer. Looking at the expansions of the concrete prisms at 365 days test age, both 40% (0.02%) and 50% (0.04%) slag mixes appear to be effective in mitigating the expansions to below 0.04%. However, the 441 days expansions indicate that the 365 days expansion value would be misleading as the expansions had increased beyond the 0.04% limit past 365 days.

In sum, slag is effective in mitigating the concrete expansions at 40% and 50% dosage in 1N NaOH exposure, while these dosages of slag do not provide adequate mitigation in presence of potassium acetate.

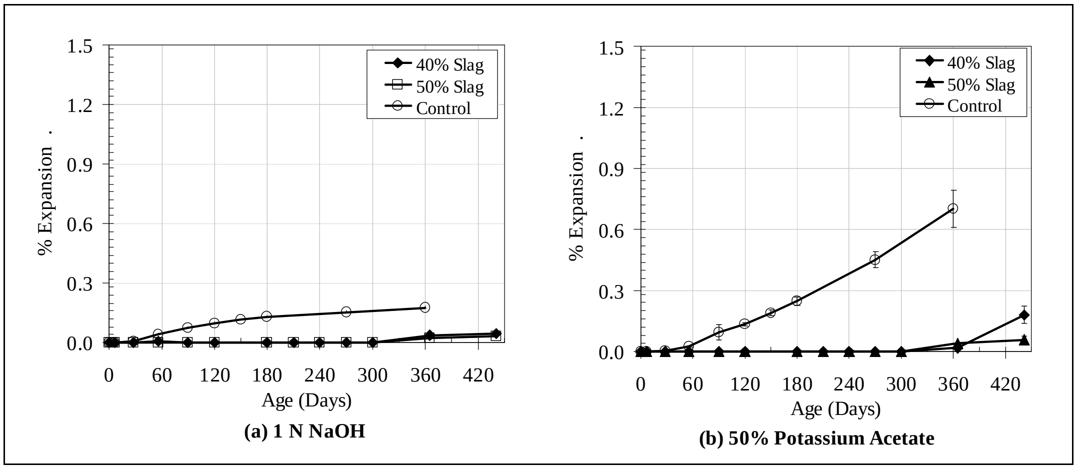



Figure 4.54 Expansions of Concrete Prisms Containing Spratt Limestone Aggregate in Modified ASTM C 1293 Tests with Slag at 40% and 50% Dosage.

<u>Influence of Modified ASTM C 1293 on DME</u>

Figure 4.55 show the changes in the DME of the concrete prisms containing Spratt aggregate in combination with slag at 40% and 50% dosage in the modified ASTM C 1293 tests.

The trend of DME results over the testing period of the modified ASTM C 1293 indicates that the DME there is no change in the DME after its initial increase at 1 month test age. However, comparing the DME results of 1N NaOH and potassium acetate (KAc) in both 40% and 50% slag mixes, it appears that the concrete prisms in potassium acetate exposure have a lower DME values compared to those in 1N NaOH. Also, the higher slag dosage at 50% imparts a higher DME to the concrete specimens compared to 40% dosage.

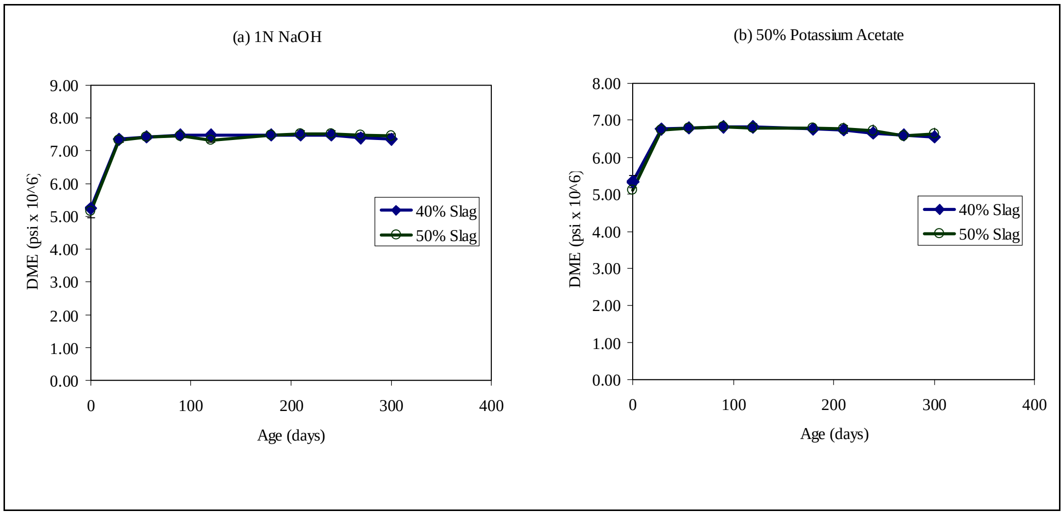



Figure 4.55 Changes in DME of Concrete Prisms Containing Spratt Limestone Aggregate in the Modified ASTM C 1293 Tests with Slag at 40% and 50% Dosage

### <u>4.6.2 Microstructure Studies- Modified ASTM C 1293 Tests Involving Spratt Limestone with Slag</u>

Figure 4.56 and 4.57 shows the visual images of concrete prisms containing Spratt limestone aggregate with slag at 40% dosage and exposed to 1N NaOH and potassium acetate respectively.

Visual images shown in figure 4.56 indicate no signs of physical distress on the surface of the concrete prisms exposed to 1N NaOH. However, the SEM micrographs shown in figure 4.58 show distress cracks within the aggregate and in the mortar matrix. These cracks provide evidence to the increase in the expansion of the concrete prisms beyond 0.04% beyond the 300 days test age. Cracking of the aggregate particles near the periphery was observed for some aggregate particles (see figure 4.58C and D). Figure 4.58(A) shows the de-bonding of an aggregate particle from the cement paste as a result of the expansion of the later.

Visual images shown in figure 4.57 show cracking of the concrete prisms exposed to potassium acetate. However, the cracks are not present all over the surface of the concrete prism and are limited to few cracks only. Examination of the concrete samples under SEM reveals signs of cracking within the aggregate particles and mortar matrix (see figure 4.59 C and D). Signs of de-bonding of aggregate and cement paste were also observed and are shown in figure 4.59 (A) and (B).

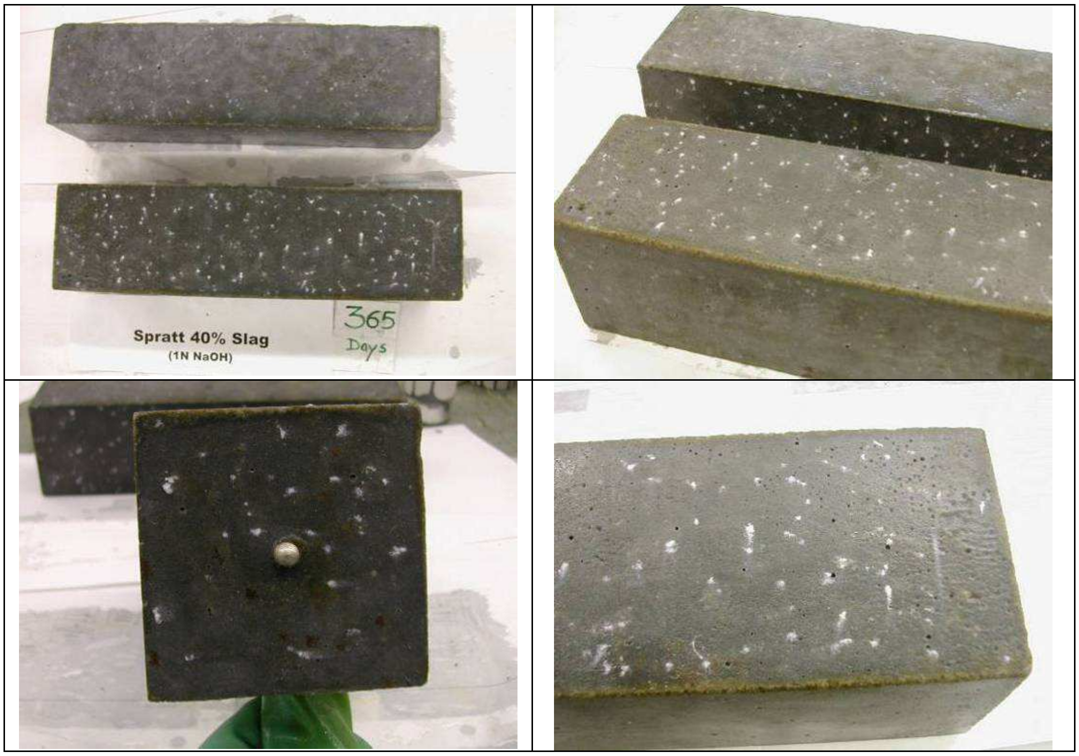



Figure 4.56 Visual Images of Concrete Prisms Containing Spratt Limestone Aggregate with 40% Slag Soaked in 1N NaOH for 365 Days

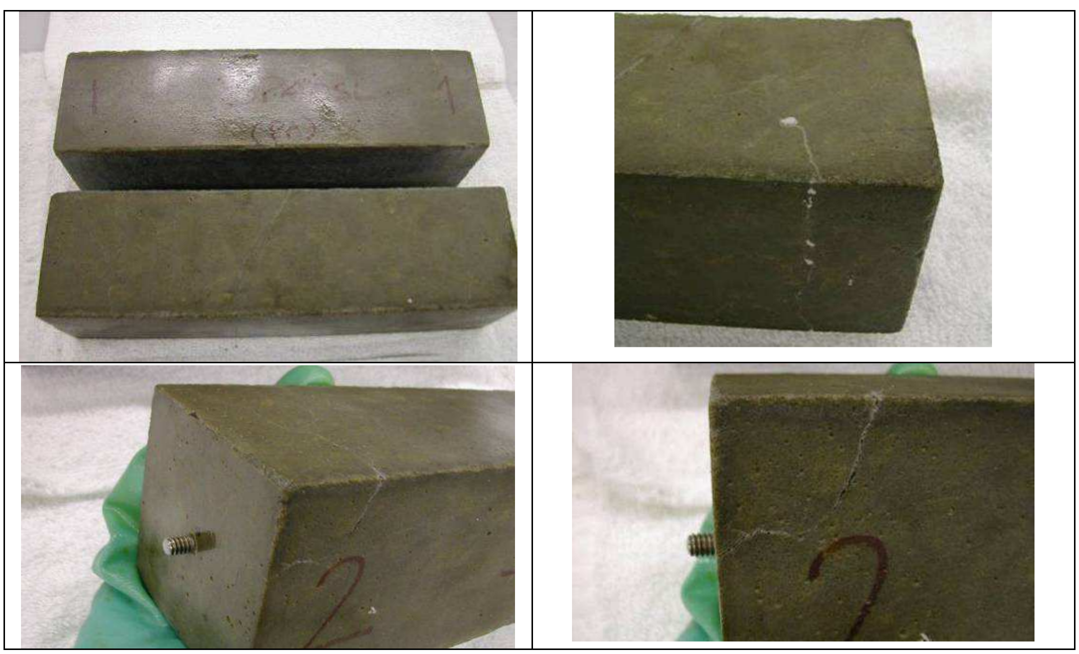


Figure 4.57 Visual Images of Concrete Prisms Containing Spratt Limestone Aggregate with 40% Slag Soaked in KAc for 365 Days

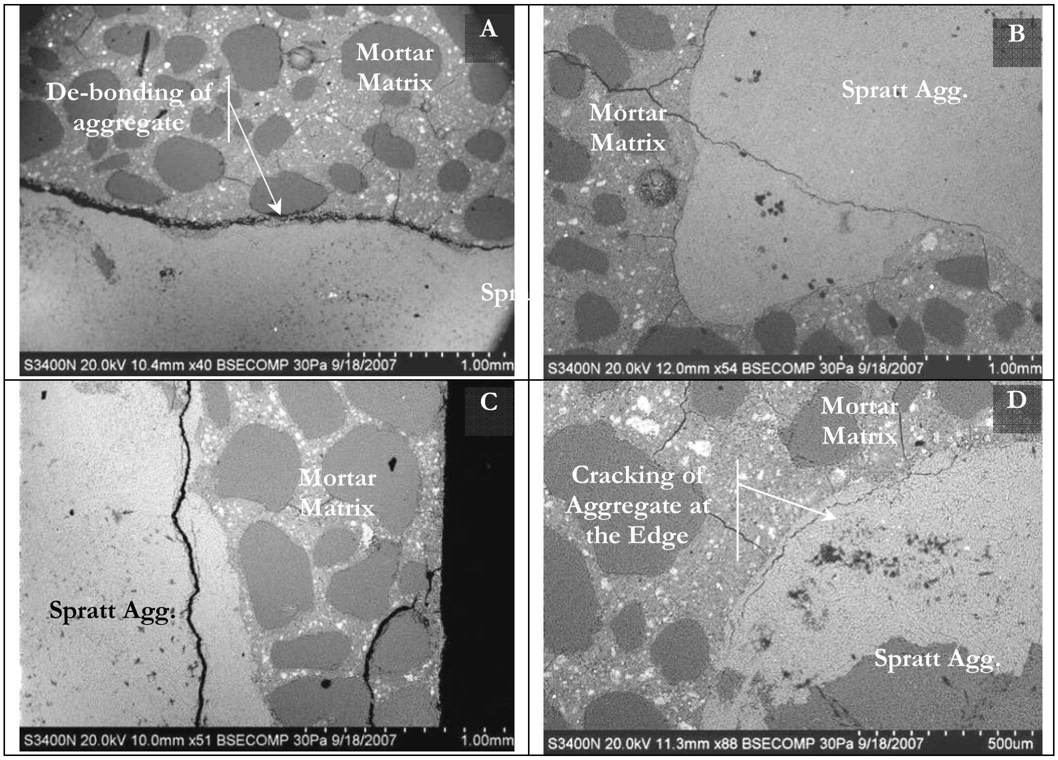



Figure 4.58 SEM Micrographs of Concrete Prism Samples Containing Spratt Limestone With 40% Slag in 1N NaOH
(A) De-bonding of Aggregate from the Cement Matrix (B) and (C) Cracking through the Aggregate into the Paste, (D) Cracking of the Aggregate Particle at the Edge.

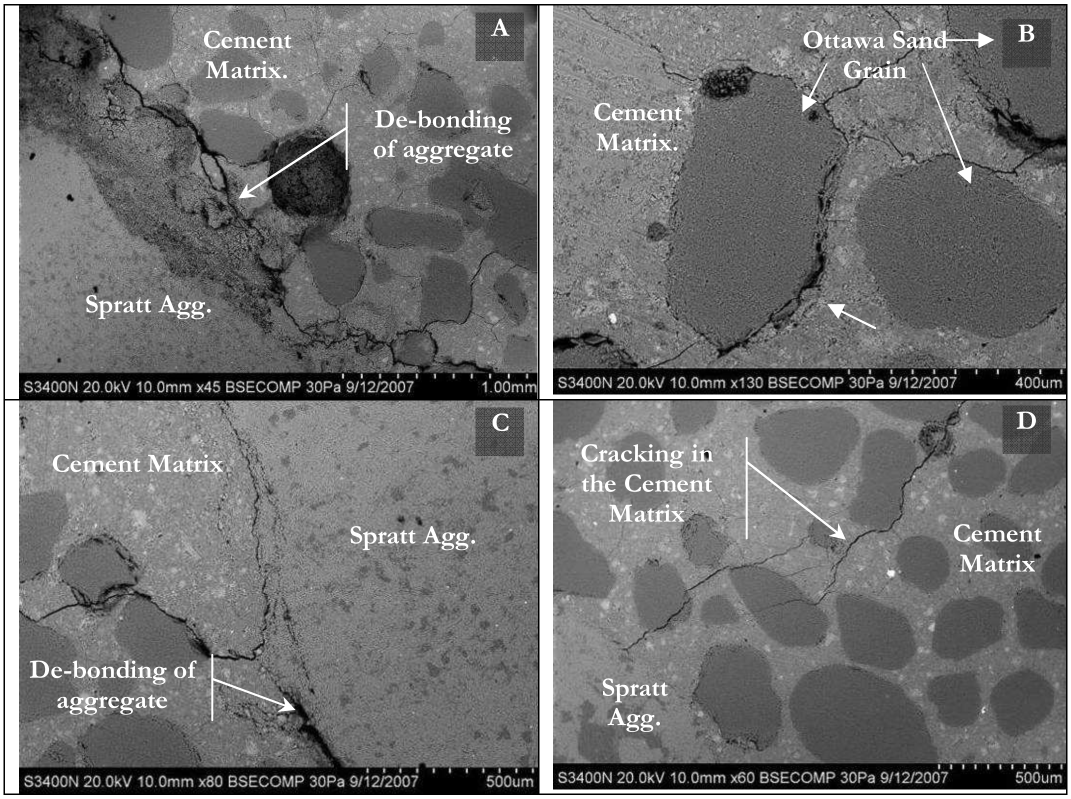



Figure 4.59 SEM Micrographs of Concrete Prism Samples Containing Spratt Limestone With 40% Slag in KAc
(A) and (C) De-bonding of Aggregate from the Cement Matrix, (B) Cracking in the Cement Matrix and De-bonding of Ottawa Sand Grain (D) Cracking in the Cement Matrix.

Figure 4.60 and 4.61 shows the visual images of concrete prisms containing Spratt aggregate with slag at 50% dosage and exposed to 1N NaOH and potassium acetate respectively.

As seen in the expansion results recorded in the modified ASTM C 1293 test, the concrete prisms containing 50% slag had expansions below 0.04% in both 1N NaOH and KAc exposure and there we no signs of cracking or physical distress as seen in the visual images. Though the SEM micrographs shown in figure 4.62 indicate the occurrence of few minor cracks within the aggregate particles, they were not significant enough to cause expansions beyond 0.04% when exposed to 1N NaOH. However, for concrete prisms exposed to potassium acetate, the SEM micrographs indicate the presence of cracks within the cement matrix and near the aggregate-paste interface (see figure 4.63).

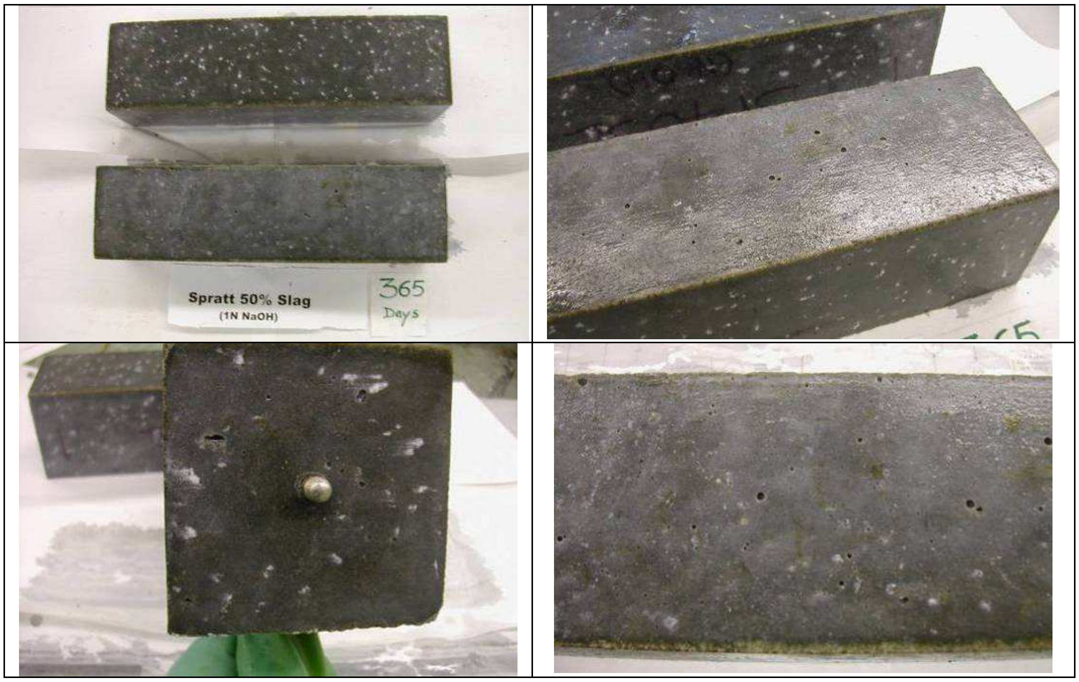



Figure 4.60 Visual Images of Concrete Prisms Containing Spratt Limestone Aggregate with 50% Slag Soaked in 1N NaOH for 365 Days

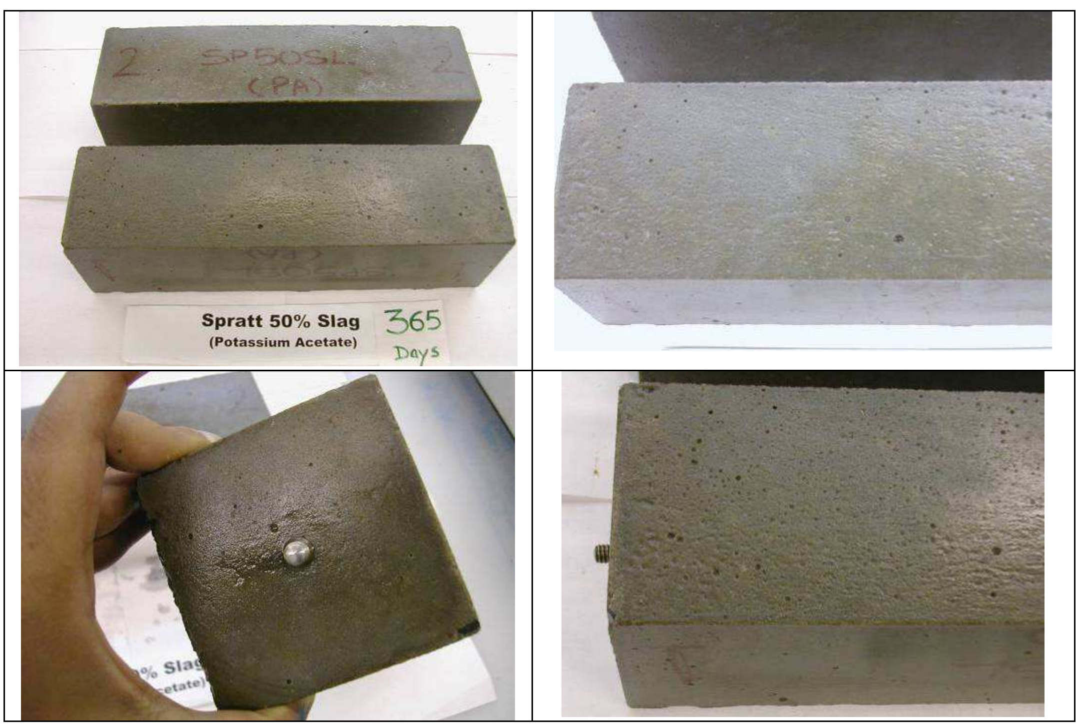



Figure 4.61 Visual Images of Concrete Prisms Containing Spratt Limestone Aggregate with 50% Slag Soaked in KAc for 365 Days

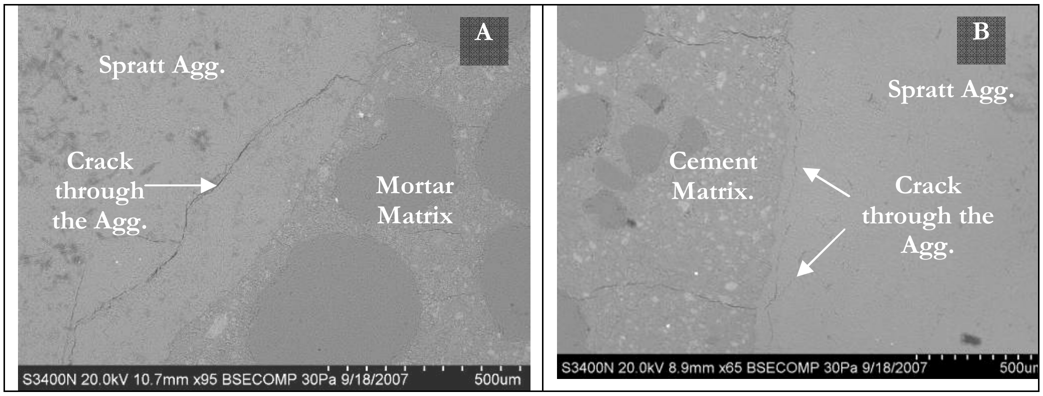



Figure 4.62 SEM Micrographs of Concrete Prism Samples Containing Spratt Limestone With 50% Slag in 1N NaOH
(A) Crack Running through the Aggregate Particle (B) Cracking in the Cement Matrix and on the Edge of the Aggregate

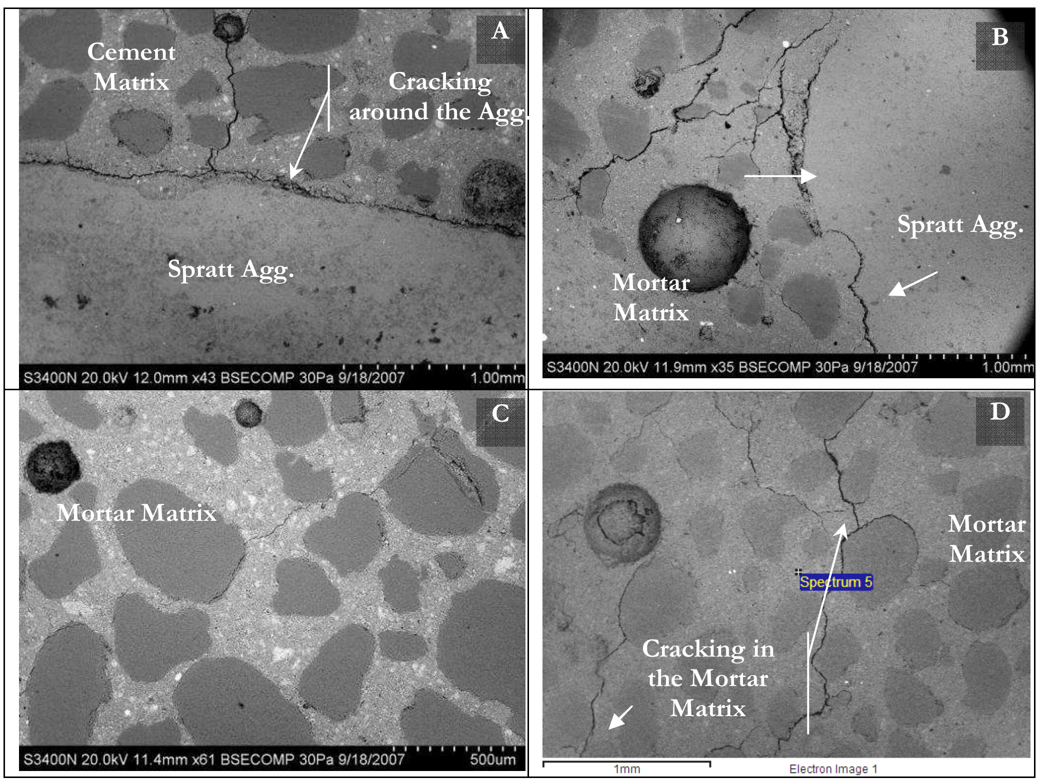



Figure 4.63 SEM Micrographs of Concrete Prism Samples Containing Spratt Limestone With 50% Slag in KAc
(A) and (B) Cracking in the Cement Matrix and Around the Aggregate Particle (C) Overall Condition of Cement Matrix (D) Cracking in the Cement Matrix in Some Areas.

New Mexico Rhyolite

Figure 4.64 show the results of the concrete prisms containing New Mexico rhyolite aggregate in combination with slag at 40% and 50% dosage in the presence of 1N NaOH and potassium acetate, respectively. The results are compared with the control concrete prisms containing no slag.

Results of the length change behavior of the concrete prisms containing slag at 40% and 50% dosage show contrasting mitigation abilities in 1N NaOH and potassium acetate deicer exposure. In 1N NaOH exposure, slag appears to be highly effective in reducing the expansions to below 0.04% at 1year test age at both 40% and 50% dosage. Contrary to this behavior, slag is not very effective in potassium acetate exposure at any of the two dosages. Though slag imparts a reduction in the expansions to less than the control expansions, it is inadequate to suppress the expansions to 0.04%.

The 40% slag concrete prisms showed signs of extensive cracking beyond the 120 days, while the 50% slag concrete prisms showed similar physical distress at 180 days.

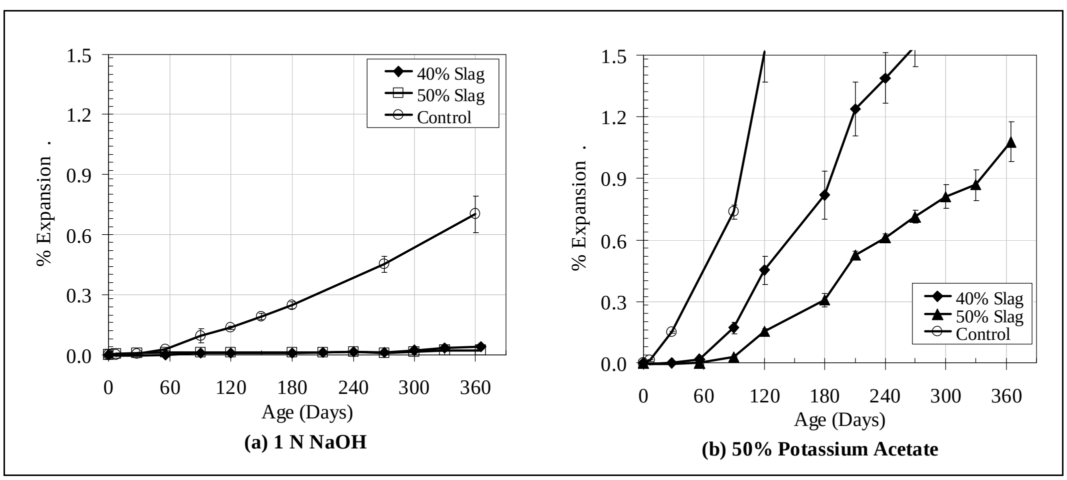



Figure 4.64 Expansions of Concrete Prisms Containing New Mexico Rhyolite Aggregate in Modified ASTM C 1293 Tests with Slag at 40% and 50% Dosage.

Influence of Modified ASTM C 1293 on DME

Figure 4.65 show the changes in the DME of the concrete prisms containing New Mexico aggregate in combination with slag at 40% and 50% dosage in the modified ASTM C 1293 tests.

The DME results of concrete prisms containing 40% and 50% slag in 1N NaOH and potassium acetate confirm the expansion results of the modified ASTM C 1293 tests. It is clearly noticeable that the concrete prisms in 1N NaOH had a constant DME over the 1 year period (after initial increase due to hardening of concrete) indicating the soundness of the concrete matrix and negligible expansion in the modified ASTM C 1293 test. Similarly, for concrete prisms in potassium acetate solution, there is a pronounced drop in the DME for 40% and 50% slag mixes indicating the loss of physical integrity of the concrete matrix due to the high expansions observed in the modified ASTM C 1293 test.

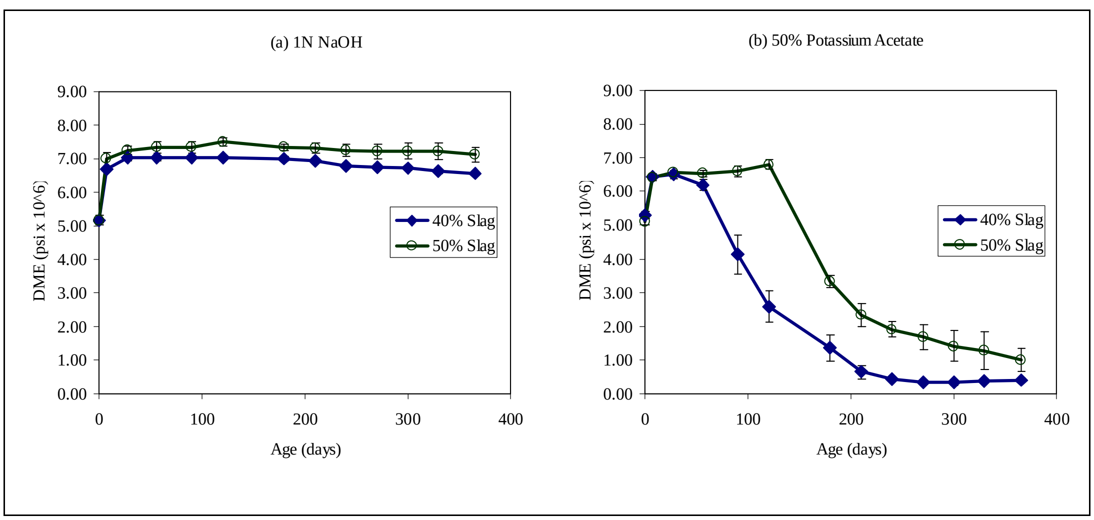



Figure 4.65 Changes in DME of Concrete Prisms Containing New Mexico Rhyolite Aggregate in the Modified ASTM C 1293 Tests with Slag at 40% and 50% Dosage

## Illinois Dolomite

Figure 4.66 show the expansions of concrete prisms containing the non-reactive Illinois dolomite aggregate I combination with 40% slag in the modified ASTM C 1293 test. The results are compared with the control concrete prisms containing no slag.

Illinois dolomite is has an established history of being non-reactive and is used as a reference aggregate in this study. This aggregate was tested in the modified ASTM C 1293 test in combination with slag to investigate the potential of slag in the presence of 1N NaOH or potassium acetate to cause deleterious expansions. Results of the length change behavior of the concrete prisms confirm that Illinois dolomite is a non-reactive aggregate and also that slag at 40% dosage does not have a negative influence on its combination with this aggregate in the presence of either 1N NaOH or potassium acetate.

Figure 4.29 show the change in DME of the concrete prisms containing 40% slag in the presence of 1N NaOH and potassium acetate (KAc). The DME results support the expansion results by showing no signs of drop in the DME since its initial increase.

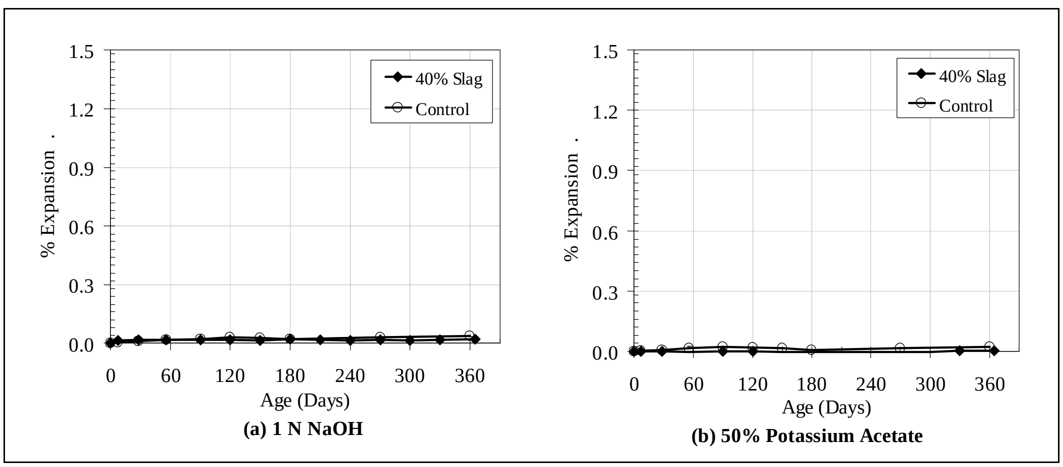



Figure 4.66 Expansions of Concrete Prisms Containing Illinois Dolomite Aggregate in Modified ASTM C 1293 Tests with Slag at 40% Dosage.

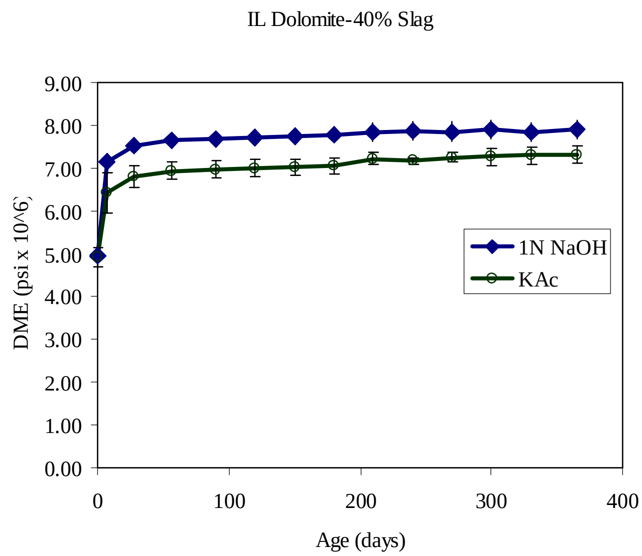



Figure 4.67 Changes in DME of Concrete Prisms Containing Illinois Dolomite Aggregate in the Modified ASTM C 1293 Tests with Slag at 40% Dosage

## 4.7 Correlations between Results of ASTM C 1293 and ASTM C 1567

This section presents a discussion on the results of the modified ASTM C 1293 tests and standard and modified ASTM C 1567 tests. Since both these tests are used to evaluate the potential for alkali-silica reactivity of aggregates, it is necessary to check the correlation between the results of the modified versions of the two tests and establish if the same correlation holds true for tests with potassium acetate deicer solution exposure.

The acceptance limit for the mortar bar test is 0.1% at 14 days and that for the standard concrete prism test is 0.04% at 1 year. For plotting the results shown in figure 4.44 and 4.45, the 14 day expansion in the standard and modified ASTM C 1567 and the 365 day expansion for the modified ASTM C 1293 test is taken for all the six fly ash mixes (Low lime fly ash-25% and 35% dosage, Intermediate lime fly ash- 25% and 35%, high lime fly ash-25% and 35%) and two slag mixes (40% and 50% dosage) for each aggregate.

4.7.1 Spratt Limestone

Figure 4.68 shows the comparison of the 14 day results of the standard and modified ASTM C 1567 mortar bar test and, the 365 day results of the modified ASTM C 1293 test for Spratt aggregate. The results are presented in a tabular form in table 4.1. Of the 8 mixes exposed to 1N NaOH, 4 (25%LL, 35% LL, 35% IL and 50%SL) conform to the acceptance limits of both the tests and the aggregate with that particular cement-fly ash/slag combination can be considered as non-reactive. 3 (40%SL, 25%HL, 35%HL) mixes with 40% slag and high lime fly ash at both the dosage levels fail to conform to the acceptance limits of both the tests. However, intermediate lime fly ash at 25% (25%IL) conform the ASTM C 1567 limit of 0.1% at 14 days, but exceed the 0.04% limit of ASTM C 1293. It is established that the results of the ASTM C 1293 tests are more valid in case of conflicting results with the mortar bar tests- ASTM C 1567 and hence the C 1293 results prevail.

Of the 8 mixes exposed to potassium acetate, low lime fly ash at 25% and 35% (25%LL, 35%LL) and intermediate lime fly ash at 35% (35%IL) conform to both ASTM C 1567 and ASTM C 1293 limits. While high lime fly ash at both 25% and 35% dosage failed the limits of both the tests. Intermediate lime fly ash at 25% (25%IL) and slag at 50% (50%SL) pass the acceptance limits of ASTM C 1567 but fail the ASTM C 1293 test. Similarly, for slag at 40% (40%SL) it passes the C 1293 test but fails the C 1567 test.

It was interesting to note that though Spratt in combination with slag at 40% dosage passes the C 1293 test (0.024%), fails to meet the test limit at a higher dosage of 50% at 365 days (0.0403%). This can be misleading because the 441 day expansions of both these test samples show that 40% slag concrete prisms expand significantly (0.18%) beyond the 365 days test limit, while the 50% slag has a gradual expansion (0.06%) at 441 days. Using the

441 day results and comparing them with the 14 day test results of the mortar bar tests; this aggregate-slag combination fails both the C 1293 and C 1567 test limits.

Table 4.2 presents the comparison of the 365 day results of C 1293 test with the 28 day expansion results of C 1567 test. The potential of reactive aggregates to expand at a later age can be seen from the results of the C 1567 tests where all the fly ash and slag mixes that passed the C 1567 test limits at 14 days in 1N NaOH exposure, appear to fail the test. This creates conflicting results between the two tests and 4 mixes (25%LL, 35% LL, 35%IL and 50%SL) have results which conform to the C 1293 test but fail the C 1567 test. However, in the case of 28 day results of potassium acetate exposure, only intermediate lime fly ash at 25% dosage failed the C 1567 test that it had previously passed at the 14 day test age.

Looking at the results of all the fly ash and slag mixes containing Spratt aggregate in 1N NaOH, none of the fly ashes or slag conforms to both the test acceptance limits of being considered non-reactive at both 14 and 28 days. In the case of results of these mixes in potassium acetate exposure, low lime fly ash at 25% and 35% dosage level and intermediate lime fly ash at 35% dosage conform to the limits of both the tests at both 14 and 28 day test age.

On performing a regression analysis between the results of C 1293 and C 1567, there seems to be a good correlation between the results in both 1N NaOH and potassium acetate exposure conditions.

Table 4.1 Comparison of the 365 Day Results of Modified ASTM C 1293 and 14 Day Results of Standard and Modified ASTM C 1567 for Spratt Aggregate

| | **Spratt- 1N** | | | **Spratt- Pot. Acetate** | | |
|---|---|---|---|---|---|---|
| | **1293M** | **1567S** | **Result** | **1293M** | **1567M** | **Result** |
| **25%LL** | Pass | Pass | Pass | Pass | Pass | Pass |
| **35%LL** | Pass | Pass | Pass | Pass | Pass | Pass |
| **25%IL** | **Fail** | **Pass** | ***Conflict*** | **Fail** | **Pass** | ***Conflict*** |
| **35%IL** | Pass | Pass | Pass | Pass | Pass | Pass |
| **25%HL** | Fail | Fail | Fail | Fail | Fail | Fail |
| **35%HL** | Fail | Fail | Fail | Fail | Fail | Fail |
| **40%SL** | Fail | Fail | Fail | **Pass** | **Fail** | ***Conflict*** |
| **50%SL** | Pass | Pass | Pass | **Fail** | **Pass** | ***Conflict*** |
| | **365 Day** | **14 Day** | | **365 Day** | **14 Day** | |

Table 4.2 Comparison of the 365 Day Results of Modified ASTM C 1293 and 28 Day Results of Standard and Modified ASTM C 1567 for Spratt Aggregate

| | **Spratt- 1N** | | | **Spratt- Pot. Acetate** | | |
|---|---|---|---|---|---|---|
| | **1293M** | **1567S** | **Result** | **1293M** | **1567M** | **Result** |
| **25%LL** | **Pass** | **Fail** | ***Conflict*** | Pass | Pass | Pass |
| **35%LL** | **Pass** | **Fail** | ***Conflict*** | Pass | Pass | Pass |
| **25%IL** | Fail | Fail | Fail | Fail | Fail | Fail |
| **35%IL** | **Pass** | **Fail** | ***Conflict*** | Pass | Pass | Pass |
| **25%HL** | Fail | Fail | Fail | Fail | Fail | Fail |
| **35%HL** | Fail | Fail | Fail | Fail | Fail | Fail |
| **40%SL** | Fail | Fail | Fail | **Pass** | **Fail** | ***Conflict*** |
| **50%SL** | **Pass** | **Fail** | ***Conflict*** | **Fail** | **Pass** | ***Conflict*** |
| | **365 Day** | **28 Day** | | **365 Day** | **28 Day** | |

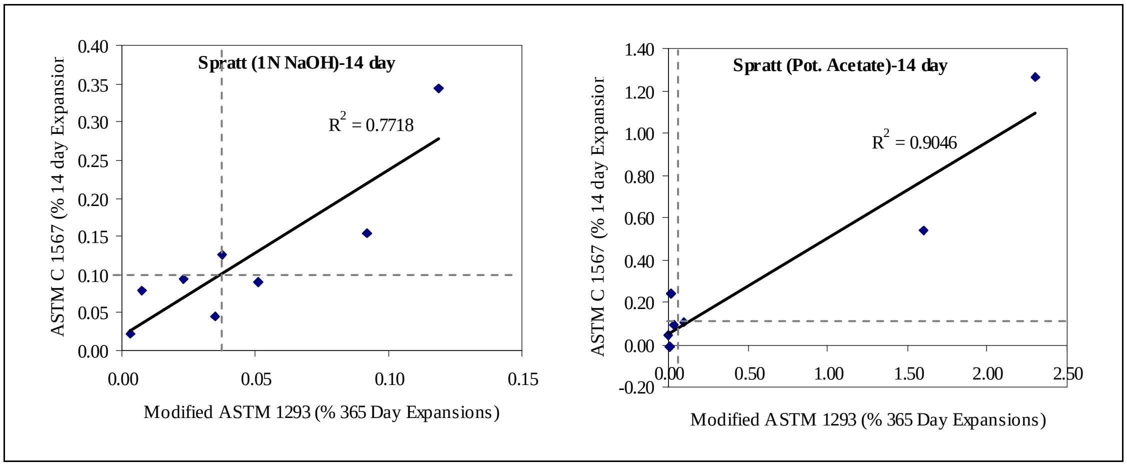



Figure 4.68 Correlation Between the Results of ASTM C 1293 at 365 days and ASTM C 1567 at 14 days Test Age for Spratt Aggregate

## 4.7.2 New Mexico Rhyolite

Figure 4.69 shows the comparison of the 14 day results of the standard and modified ASTM C 1567 mortar bar test and, the 365 day results of the modified ASTM C 1293 test for New Mexico aggregate. The results are presented in a tabular form in table 4.3.

Of the 8 mixes of fly ash and slag in 1N NaOH exposure, only intermediate lime fly ash at 35% dosage (35%IL) conform to the acceptance limit of both the tests. Mixes with low lime fly ash at 35% dosage, high lime fly ash at both 25% and 35% dosage, and slag at 40% dosage appear to fail in both the tests. However, low lime fly ash at 25% and slag at 50% pass the C 1293 test but fail the C 1567 test. It should be noted that except for intermediate lime fly ash at 35% dosage none of the fly ash or slag mixes passed the C 1567 test in 1N NaOH exposure.

Of the 8 mixes exposed to potassium acetate, none of the mixes of fly ash and slag passed both the C 1293 and C 1567 test. All the mixes failed in the C 1293 test at the 365 day test age expansion limit, whereas only low lime fly ash at 25% and 35%, and slag at 50% passed the C 1567 test at 14 days.

A comparison of the 365 day results of the C 1293 test with the 28 day results of C 1567 test in both 1N NaOH and potassium acetate exposure is presented in table 4.4. At 28 day test age, out of the four mixes (1 in 1N NaOH and 3 in potassium acetate) that passed the C 1567 test at 14 days test age, only two mixes (Low lime fly ash at 35% and Slag at 50% in potassium acetate) had expansions below the acceptance limit even at 28 days and passed the C 1567 test.

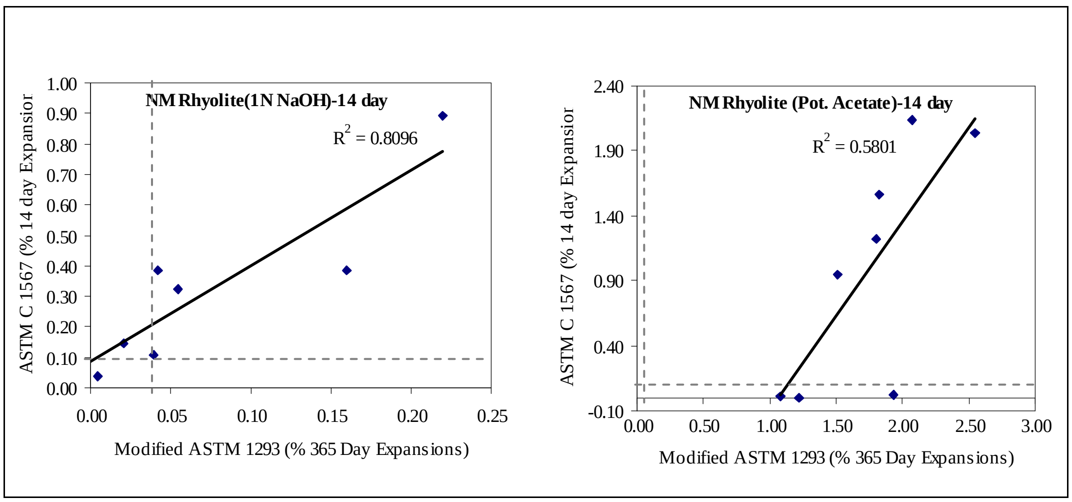



Figure 4.69 Correlation Between the Results of ASTM C 1293 at 365 days and ASTM C 1567 at 14 days Test Age for NM Rhyolite Aggregate

Table 4.3 Comparison of the 365 Day Results of Modified ASTM C 1293 and 14 Day Results of Standard and Modified ASTM C 1567 for New Mexico Aggregate

| | **New Mexico- 1N** | | | **New Mexico- Pot. Acetate** | | |
|---|---|---|---|---|---|---|
| | **1293M** | **1567S** | **Result** | **1293M** | **1567M** | **Result** |
| **25%LL** | **Pass** | **Fail** | ***Conflict*** | **Fail** | **Pass** | ***Conflict*** |
| **35%LL** | **Pass** | **Fail** | ***Conflict*** | **Fail** | **Pass** | ***Conflict*** |
| **25%IL** | Fail | Fail | Fail | Fail | Fail | Fail |
| **35%IL** | Pass | Pass | Pass | Fail | Fail | Fail |
| **25%HL** | Fail | Fail | Fail | Fail | Fail | Fail |
| **35%HL** | Fail | Fail | Fail | Fail | Fail | Fail |
| **40%SL** | Fail | Fail | Fail | Fail | Fail | Fail |
| **50%SL** | **Pass** | **Fail** | ***Conflict*** | **Fail** | **Pass** | ***Conflict*** |
| | **365 Day** | **14 Day** | | **365 Day** | **14 Day** | |

Table 4.4 Comparison of the 365 Day Results of Modified ASTM C 1293 and 28 Day Results of Standard and Modified ASTM C 1567 for New Mexico Aggregate

| | **New Mexico- 1N** | | | **New Mexico- Pot. Acetate** | | |
|---|---|---|---|---|---|---|
| | **1293M** | **1567S** | **Result** | **1293M** | **1567M** | **Result** |
| **25%LL** | **Pass** | **Fail** | ***Conflict*** | Fail | Fail | Fail |
| **35%LL** | **Pass** | **Fail** | ***Conflict*** | **Fail** | **Pass** | ***Conflict*** |
| **25%IL** | Fail | Fail | Fail | Fail | Fail | Fail |
| **35%IL** | Fail | Fail | Fail | Fail | Fail | Fail |
| **25%HL** | Fail | Fail | Fail | Fail | Fail | Fail |
| **35%HL** | Fail | Fail | Fail | Fail | Fail | Fail |
| **40%SL** | Fail | Fail | Fail | Fail | Fail | Fail |
| **50%SL** | **Pass** | **Fail** | ***Conflict*** | **Fail** | **Pass** | ***Conflict*** |
| | **365 Day** | **28 Day** | | **365 Day** | **28 Day** | |

Looking at the results of all the fly ash and slag mixes containing New Mexico aggregate in 1N NaOH, none of the fly ashes or slag conforms to both the test acceptance limits of being considered non-reactive at both 14 and 28 days. In the case of results of these mixes in potassium acetate exposure, low lime fly ash at 25% and 35% dosage level and intermediate lime fly ash at 35% dosage conform to the limits of both the tests at both 14 and 28 day test age.

On performing a regression analysis between the results of C 1293 and C 1567 tests, there seems to be a good correlation between the results in 1N NaOH exposure. However, there is not a good correlation between the results of the two tests in potassium acetate exposure.

### 4.8 Influence of Chemical Composition of Fly Ash on ASR Mitigation

It is an established fact that the chemistry of the fly ash is one of the most important criteria in distinguishing between fly ashes for their effectiveness in mitigating ASR. Different approaches have been used by researchers to identify the oxides or a combination of the oxides in the chemical composition of the fly ash that control the expansions of the mortar bars in the Standard ASTM C 1567 test (Touma et al. 2001, Detwiler 2003, Thomas and Shehata 2004, Malvar et al.2006). This section presents the analysis of the various oxide constituents of the fly ash and its influence on the expansions of mortar bars in the standard and modified ASTM C1567 test.

For this analysis, the results of the standard and modified ASTM C 1567 mortar bar test using Spratt limestone aggregate in combination with 15 fly ashes at 25% cement replacement level were used. The chemical constituents of the fly ash and cement were weighted based on their relative percentages (by weight) in the cement-fly ash blend. The following methods are used to find correlations between the mortar bar expansions and the chemical constituents of the fly ash:

- Influence of individual chemical constituent as a sum of the fly ash-cement blend (%CaO, %MgO, $\%SO_3$, $\%SiO_2$, $\%Al_2O_3$, $\%Fe_2O_3$, alkalis expressed as $Na_2O_{eq}= Na_2O+0.658\ K_2O$) on the mortar bar expansions at 14 days. Since there is only one type of cement used in this study and the cement replacement level of fly ash for this analysis is constant (25%), the only variable will be the oxide content as a factor of the fly ash type. Hence, the influence of the total oxide component (fly ash-cement blend) on the expansions of the mortar bars will be mainly due to the change in the oxide composition of the different fly ashes.

- Influence of combination of chemical constituents in the fly ash-cement blend that promote expansion. Since CaO has been recognized as one of the most damaging chemical constituents in terms of its influence on ASR expansions, the other deleterious constituents such as MgO, $SO_3$ and alkalis (represented as $Na_2O_{eq}$= $Na_2O$+0.658 $K_2O$) were replaced by their CaO molar equivalents as shown in the following equation:

$$\mathbf{CaO_{eq} = CaO + 0.905\ Na_2O_{eq} + 1.391\ MgO + 0.70\ SO_3}$$

- Influence of combination of chemical constituents in fly ash-cement blends that reduce expansion. Like CaO is recognized for it expansion promoting ability, $SiO_2$ is considered to be the most beneficial constituent in preventing expansion. Hence the benign oxides (i.e. that reduce or do not affect expansion) such as $Al_2O_3$ and $Fe_2O_3$ were replaced by their $SiO_2$ molar equivalents as shown in the following equation:

$$\mathbf{SiO_{2\,eq} = SiO_2 + 0.589\ Al_2O_3 + 0.376\ Fe_2O_3}$$

- Influence of the $\mathbf{CaO_{eq}/\ SiO_{2\,eq}}$ ratio of the fly ash-cement blend on the mortar bar expansions.
- Influence of the $\mathbf{CaO/(SiO_2)^2}$ ratio of the fly ash-cement blend on the mortar bar expansions (based on Thomas et al. 2004).

4.8.1 Influence of Individual Fly Ash Chemical Constituent on Mortar Bar Expansions

Figure 4.70 presents the correlations between the expansion promoting chemical constituents of the fly ash -cement blend and the mortar bar expansions in the standard (1N NaOH) and modified (Potassium acetate) ASTM C 1567 test at 14 days.

- Calcium Oxide (CaO)

Since the cement CaO content and the cement replacement by fly ash (25%) is constant for all the blends, the cementitious CaO variation is mostly due to the lime content of the fly ash only

As seen in figure 4.70, there seems to be a good correlation between the lime content of the cement-fly ash blend and the mortar bar expansions at 14 days. The CaO content for the fly ashes varied from 1.27% to 29.85%, and 61.84% for the cement. The noticeable difference between the expansion trends in 1N NaOH and potassium acetate is the degree of expansion with the increase in the %CaO content of the blend. In 1N NaOH the increase in expansion with an increase in the CaO content seems more gradual with increased expansions observed beyond 50% CaO content of the blend. Contrary to this behavior, the increase in the expansions for the mortar bars exposed to potassium acetate is more sudden for lime contents beyond 52%..

- Sulfur Trioxide ($SO_3$)

From figure 4.70 it appears that there is a strong positive correlation between the mortar bar expansions and the $SO_3$ content of the cement-fly ash blend, indicating that as the $SO_3$ content of the blend increases so does the expansions.

However, there is a striking similarity between the degree of increase in the expansions on increasing the CaO and the $SO_3$ content. In both the cases, the increase in the expansions on increasing the CaO or $SO_3$ is gradual for 1N NaOH exposure samples, and sudden for mortar bars exposed to potassium acetate. The expansions increase abruptly for $SO_3$ contents beyond 3.60% and up to 3.80%.

It should be noted that all the fly ashes used in this study have $SO_3$ contents within the ASTM C 618 specified limit (<5.0%) for Class C and F fly ashes.

- <u>Magnesium Oxide (MgO)</u>

There seems to be a moderate correlation between the MgO content and the 14 day expansions of the mortar bars in both 1N NaOH and potassium acetate exposure. Similar to what was observed in the correlations for CaO and SO3, the three Class C fly ashes having lime content above 27% appear to increase the expansions dramatically in combination with $SO_3$ and MgO. From figure 4.70, it appears that there is a sudden increase in the expansions beyond 3.1% MgO content. However, a moderate to weak correlation is expected for MgO because it occurs in a non-crystalline form or in the form of a non-expansive melilite phase in the fly ash (Helmuth 1987).

All the fly ashes used in this study contain MgO within the ASTM C 618 specified limit of 5%.

- <u>Alkalis ($Na_2O$ and $K_2O$ represented in terms of $Na_2O$)</u>

There appears to be a poor correlation between the alkali content of the cement-fly ash blend and the mortar bar expansions. This finding is in agreement with the findings from previous studies in which the alkali content of the cement was not found to be a factor affecting the expansions in the standard and modified ASTM C 1260 tests (Sompura2006).

Figure 4.71 presents the correlations between the expansion-reducing chemical constituents of the fly ash and cement blend and the mortar bar expansions in the standard (1N NaOH) and modified (Potassium acetate) ASTM C 1567 test at 14 days.

- Silicon Dioxide ($SiO_2$)

Silicon dioxide involves in the pozzolanic reaction with the calcium hydroxide produced during the hydration reactions of cement and forms a cementitious product. It is due this reason that the Class F fly ashes that have a higher SiO2 content are more effective in reducing the expansions.

Figure 4.71 indicates that there is a strong inverse correlation between the SiO2 content of the cement-fly ash blend and the mortar bar expansions. This correlation is more pronounced in the case of mortar bars exposed to 1N NaOH than for mortar bars exposed to potassium acetate. This also points out that for fly ash to be effective enough in reducing the expansions to below 0.1% at 14 days and at 25% dosage, the SiO2 content of the fly ash should be above 25%for 1N NaOH exposure, and above 26% for potassium acetate exposure.

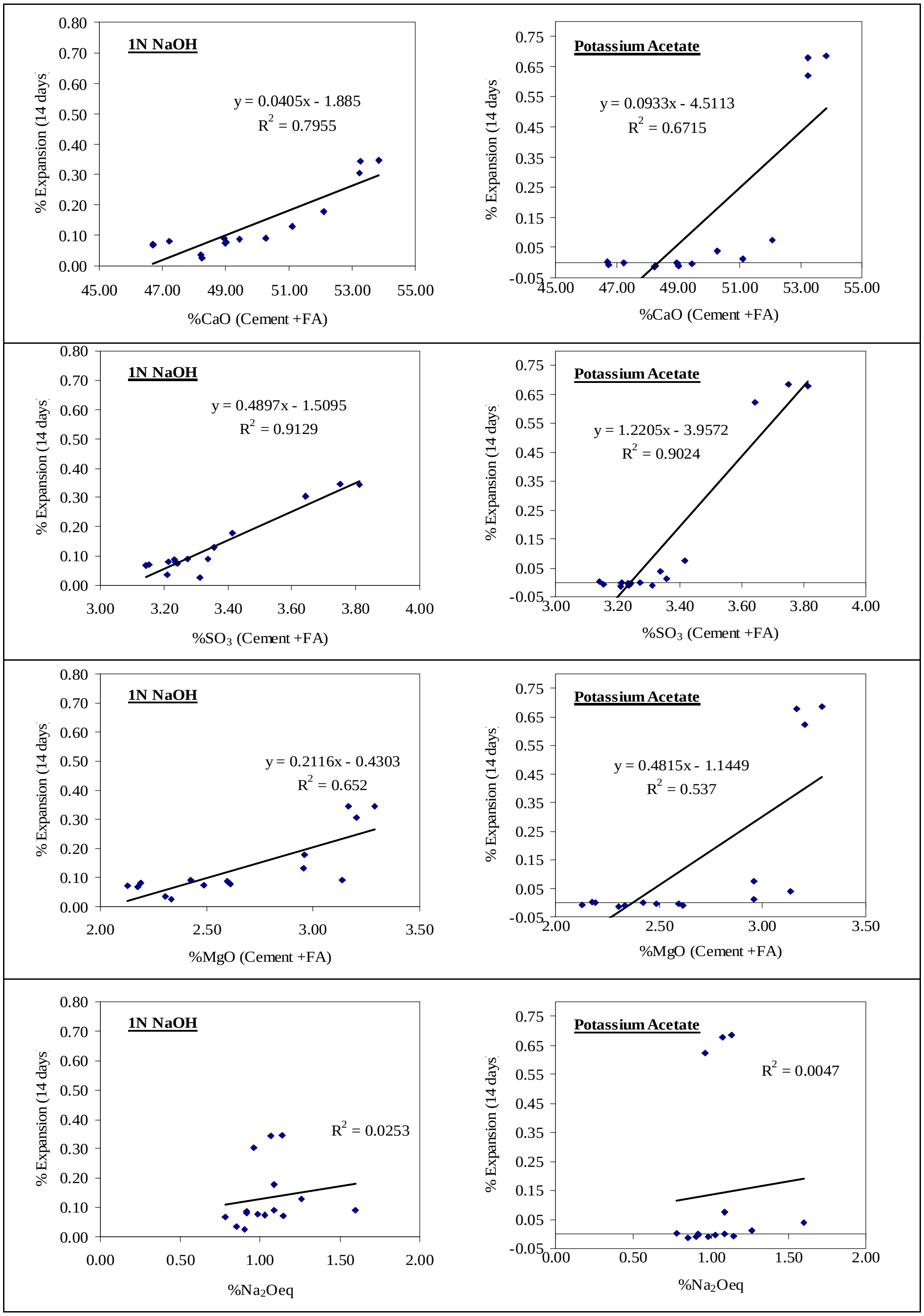



Figure 4.70 Correlations between the Expansion Promoting Chemical Constituents of Fly Ash and Mortar Bar Expansions in Standard and Modified ASTM C 1567 Test at 14 days at 25% Fly Ash Dosage.

- Aluminum Trioxide ($Al_2O_3$)

Alumina too imparts pozzolanicity to the cement-fly ash matrix, but it is the combination of $SiO_2$, $Al_2O_3$ and $Fe_2O_3$ that shows a good relation to the effectiveness of a fly ash.

From figure 4.71 it appears that there is a weak inverse correlation between the mortar bar expansions and the alumina content of the cement-fly ash blend.

- Iron Oxide ($Fe_2O_3$)

Iron oxide is present as a non-reactive phase in the fly ash in the form of hematite and magnetite. Hence, it might not contribute to the expansion reducing effect of the fly ash.

Figure 4.71 indicates that there is a poor inverse relation between the mortar bar expansions and the iron oxide content of the cement-fly ash blend.

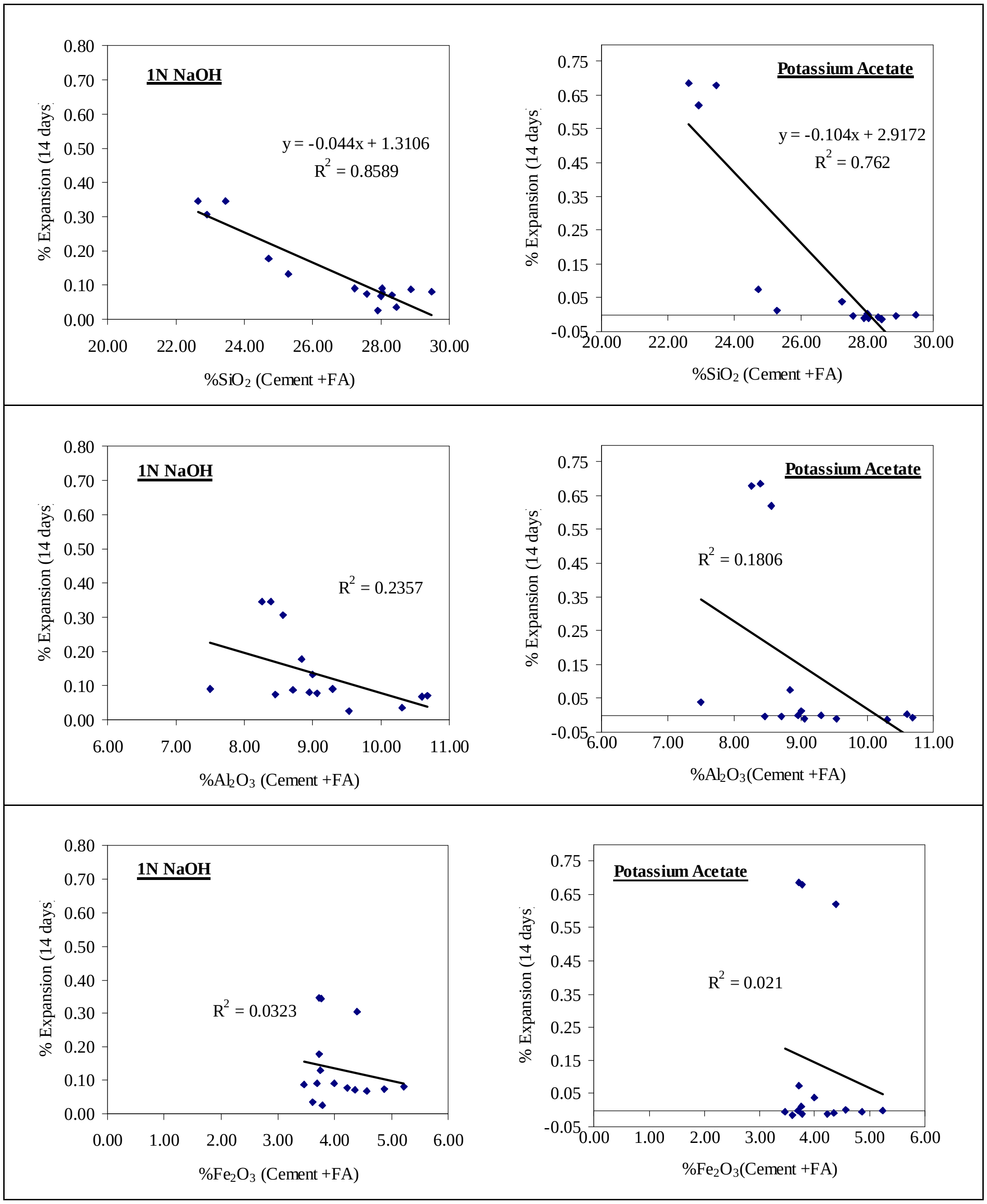



Figure 4.71 Correlations between the Expansion Reducing Chemical Constituents of Fly Ash and Mortar Bar Expansions in Standard and Modified ASTM C 1567 Test at 14 days at 25% Fly Ash Dosage.

4.8.2 Influence of Fly Ash Chemical Constituent Combinations on Mortar Bar Expansions

Figure 4.72 show the correlation of the combination of the three expansion reducing constituents of the cement-fly ash blend, and the expansion of the mortar bars at 14 days.

Influence of Combination of Chemical Constituents Reducing Expansion

Here, the combination of the expansion reducing constituents has been done in two ways to explore which combination yields a better correlation. The first correlation is done by simple adding up the $SiO_2$, $Al_2O_3$ and $Fe_2O_3$ of the cement-fly ash blend and comparing them to the mortar bar expansions at 14 days. In the second correlation, all the three oxides are converted to represent the molar equivalent of $SiO_2$ and this equivalent sum is called $SiO_{2eq}$ Looking at both the correlations, it appears that replacing the oxides by the molar equivalents of $SiO_2$ gives a better correlation with the expansions of the mortar bars than simply taking the sum of the three oxides.

There is a good inverse relationship between the 14 day expansions and the SiO2eq indicating that the cement-fly ash blend becomes more effecting in reducing the expansions if the $SiO_2$eq value is above 32%.

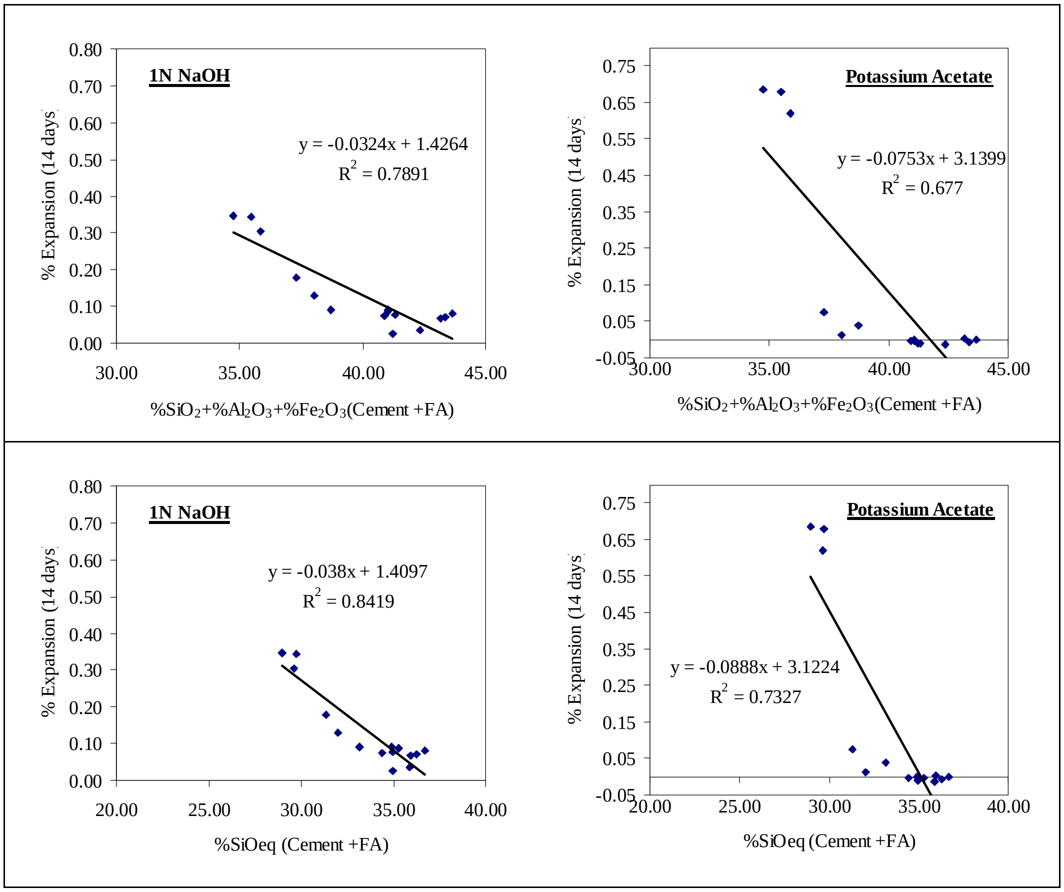



Figure 4.72 Correlations between the Combination of Expansion Reducing Chemical Constituents of Fly Ash and Mortar Bar Expansions in Standard and Modified ASTM C 1567 Test at 14 days at 25% Fly Ash Dosage.

<u>Influence of Combination of Chemical Constituents Promoting Expansion</u>

Figure 4.73 shows the correlation between the calcium oxide equivalent (CaOeq) and the mortar bar expansions at 14 days. It appears that there is a strong relationship between the two factors and increasing the content of CaO, MgO, $SO_3$ and the alkalis ($Na_2Oeq$) leads to an increase in the expansions.

On correlating the CaOeq/$SiO_2$eq ratio to the 14 day mortar bar expansions (shown in figure 4.74) it appears that there is a significant inverse relationship between this ratio and the mortar bar expansions.

On correlating the $CaO/(SiO_2)^2$ ratio (based on Thomas et al.2004),it appears that there is a significant inverse relationship between this ratio and the 14 day expansions. Also, this ratio gives a better correlation when compared to the correlation generated using the $CaOeq/SiO_2eq$ ratio.

From these two ratio's it appears that to achieve a 14 day expansion of less than 0.1% using fly ash at 25% dosage, the $CaOeq/SiO_2eq$ should be 1.50 or less for 1N NaOH, and 1.80 or less for potassium acetate exposure; and the $CaO/(SiO_2)^2$ ratio should be 0.065 or less for 1N NaOH and 0.085 or less for potassium acetate exposure.

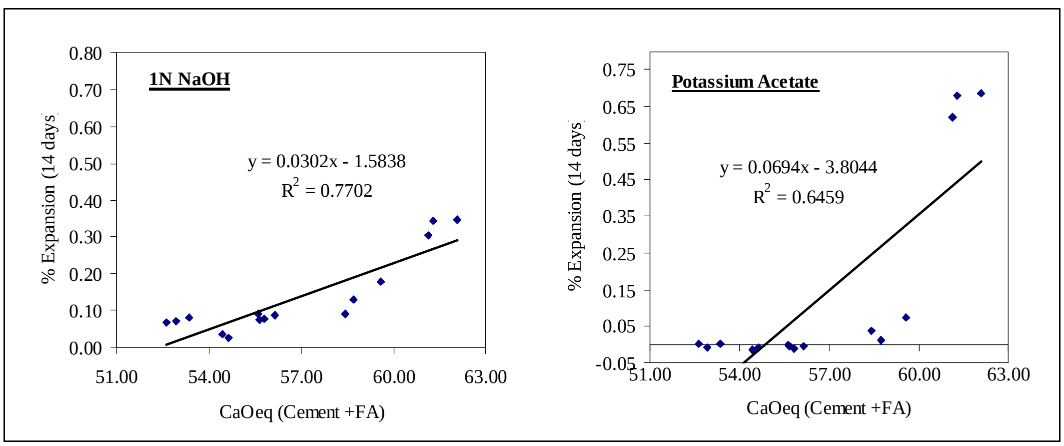



Figure 4.73 Correlations between the Combination of Expansion Promoting Chemical Constituents of Fly Ash and Mortar Bar Expansions in Standard and Modified ASTM C 1567 Test at 14 days at 25% Fly Ash Dosage.

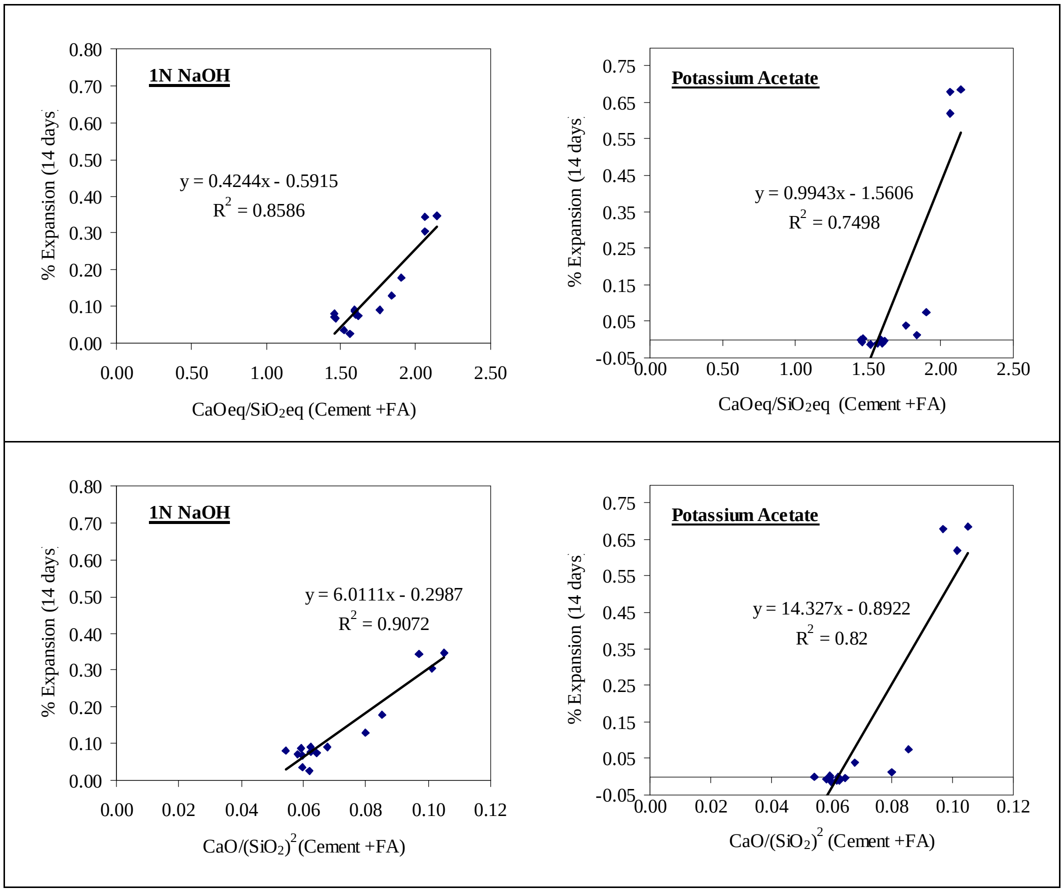



Figure 4.74 Effect of $CaOeq/SiO_2eq$ ratio and $CaO/(SiO_2)^2$ ratio on Mortar Bar Expansions in Standard and Modified ASTM C 1567 Test at 14 days at 25% Fly Ash Dosage.

## 4.9 Statistical Analyses

This section presents the results of the statistical analyses performed on the results of the standard and modified ASTM C 1567 (mortar bar tests) and the modified ASTM C 1293 (concrete prism tests) and find out if the differences between these results are statistically significant. The data gathered in this research was analyzed using Statistical Analysis System (SAS) and Microsoft Excel software packages.

SAS was used to conduct analysis of variance (ANOVA) tests to determine if significant differences existed within the sample means of fly ashes and slag at different dosages and with different aggregate types. The null hypothesis (*Ho*) for the ANOVA tests was that all sample means are equal. From the ANOVA tests, the F-test statistic value ($F_{observed}$) is calculated and is compared to the $F_{crit}$ (F critical) value. The $F_{crit}$ is based on the degrees of freedom and the level of significance ($\alpha$) which was 0.05 ($\alpha = 0.05$) for this study. If the F value ($F_{observed}$) obtained from the ANOVA calculations is greater than the $F_{crit}$ value, then the *Ho* is rejected, indicating that there is insufficient evidence to conclude that all the sample means are equal.

Once it is established that not all sample means are equal, the next step was to determine if the differences between the population means are significantly different or statistically different. This was done by determining the least significant difference (LSD) which is a value that is the observed difference between two population means to conclude if those means are statistically different or not.

To complement the results presented in sections 4.2 thru 4.6, correlations between the variables and expansions were determined by performing a regression analysis on the results data. The variables were fly ash type, fly ash dosage, slag dosage, aggregate type and soak solution (1N NaOH or KAc), while the percent expansion at 14 days (365 days for concrete prism tests) for a particular aggregate was kept constant.

In the following sections the results of the LSD tests are discussed and the tables are presented in a matrix format to show if the difference between two variables is statistically significant or not. This is done by using alphabets 'X' and 'S' where, 'X' represents a statistically significant difference between the two factors and, 'S' indicates that the difference between the two factors in comparison is similar or statistically not significant. The tables of the LSD matrices are presented in Appendix-F.

4.9.1 Influence of Fly Ash Dosage and Fly Ash Type on Expansions of Mortar Bar Tests

*Spratt Limestone*

Based on the LSD grouping (presented in Table F.1 in Appendix), it was evident that the differences between the expansions of the mortar bars irrespective of fly ash used are similar for 15% and 25% dosage in both 1N NaOH and potassium acetate exposure. This indicates that 15% and 25% fly ash dosage have the same response in terms of length change expansions when exposed to 1N NaOH and potassium acetate.

Contrary to this, the difference between 15%, and 35%, 25% and 35% 15% and 15%, 25% and 35% with 0% (control) is significant. This difference can be observed from the mean expansions of the mortar bars with these fly ash dosages as shown in figure 4.75.

Analyzing the results with regards to the influence of fly ash type on the mortar bar expansions irrespective of the effect of fly ash dosage it appears that there is a significant difference between the three types of fly ashes and the control specimens exposed to 1N NaOH. However, in potassium acetate exposure, low lime fly ash (LL) and intermediate lime fly ash (HL) have a similar response to the mortar bar expansions. This can be noticed from the similar levels of expansions for LL and IL fly ashes in figure 4.75.

Figure 4.75 show the linear regression analysis on the 14 day expansions of the mortar bars with all the three fly ashes and its relation to the fly ash dosage. Based on the coefficient of determination values, there seems to be a good inverse correlation between the 14 day expansions of the mortar bars and the fly ash dosage for low lime and intermediate lime fly ashes in both 1N NaOH and potassium acetate. However, there is a moderate correlation between high lime fly ash dosage and the mortar bar expansions in both potassium acetate and 1N NaOH. This can be attributed to the high expansions observed at 25% dosage followed by a significant drop in the expansions at 35% dosage

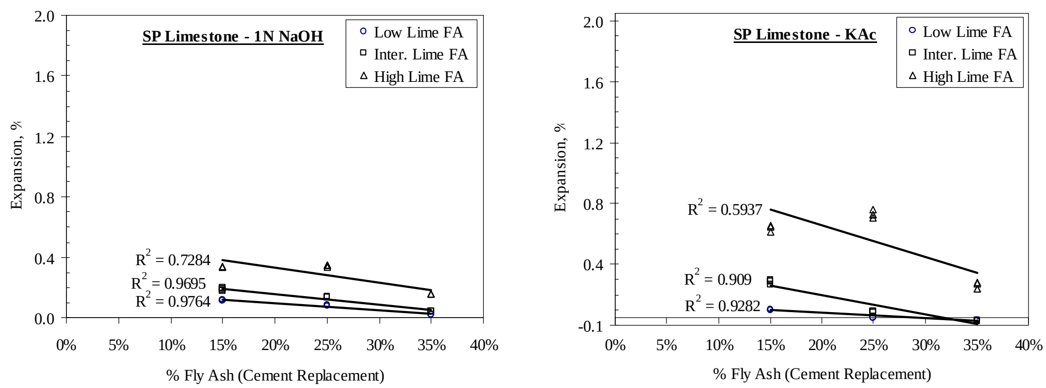



Figure 4.75 Correlations between the 14 Day Mortar bar Expansion, Fly Ash Dosage and Fly Ash Type in 1N NaOH and Potassium Acetate Exposure for SP Limestone Containing Mortar Bars.

*New Mexico Rhyolite*

Observing the expansions plotted in figure 4.76 and the LSD grouping (table F.2 in Appendix), it is evident that the differences in the expansion results, at all the three fly ash dosages and for all the three fly ashes in 1N NaOH exposure, are statistically significant. However, for mortar bars exposed to potassium acetate at 15% and 25% dosage have similar effectiveness in reducing the expansions. High lime and intermediate lime fly ashes have similar expansions when compared to control, indicating that both these fly ashes (irrespective of the dosage used) are ineffective in mitigating the expansions, and hence their differences too are statistically not significant.

Based on the regression analysis of the expansion results, it can be seen that there is a good inverse relationship between the percent expansion and fly ash dosage for a particular type of fly ash. This indicates that as the fly ash dosage increases, the mortar bar expansion reduces. This correlation is moderate for high lime fly ash in 1N NaOH because of the variability of the results.

It should be noted that there is a positive but poor correlation between the fly ash dosage and expansion for high lime fly ash containing mortar bars in potassium acetate. This indicates that as the dosage of high lime fly ash increases, so does the expansions

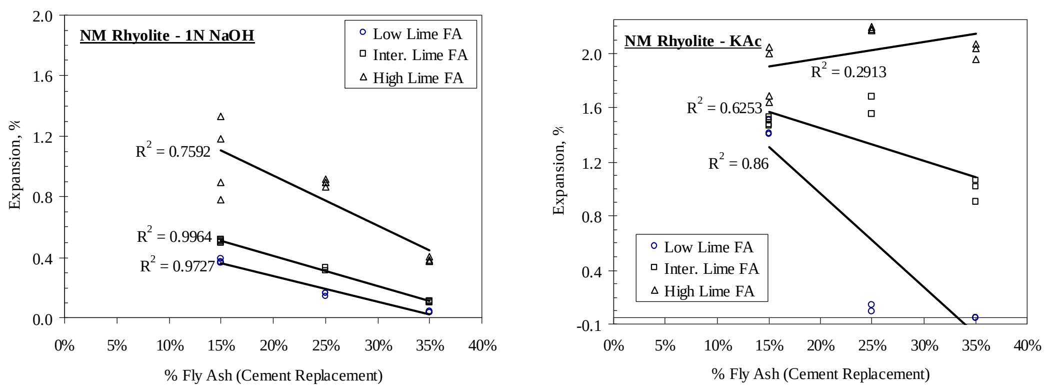



Figure 4.76 Correlations between the 14 Day Mortar bar Expansion, Fly Ash Dosage and Fly Ash Type in 1N NaOH and Potassium Acetate Exposure for NM Rhyolite Containing Mortar Bars.

### *North Carolina Argillite*

Based on the LSD grouping (table F.3 in Appendix), difference between 15% and 25% fly ash dosage in both 1N NaOH and potassium acetate is statistically insignificant, indicating a similar level of effectiveness in reducing the expansions. However, the difference between control (0%) and the three dosages (15%, 25% and 35%) is significant. With regards to the influence of fly ash type, low lime and high lime fly ash have a similar response in reducing the expansions and hence the difference between them is statistically not significant.

The correlations between the fly ash dosage and the 14 day expansions are presented in figure 4.77 and it reveals the same trend as seen in SP limestone aggregate results, with good correlation seen in low lime and intermediate lime fly ash mortar bar expansions and moderate correlation for high lime fly ash irrespective of the exposure condition (1N NaOH or potassium acetate).

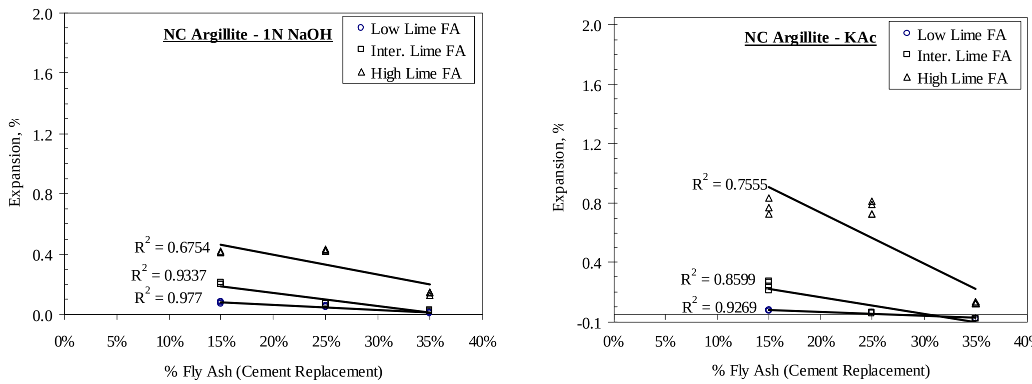



Figure 4.77 Correlations between the 14 Day Mortar bar Expansion, Fly Ash Dosage and Fly Ash Type in 1N NaOH and Potassium Acetate Exposure for NC Argillite Containing Mortar Bars.

*South Dakota Quartzite*

Based on the LSD grouping (table F.4 in Appendix) the differences between the three dosages and the control are significant in both 1N NaOH and potassium acetate irrespective of the fly ash type. With regards to the type of fly ash and its influence on 14 day expansions, high lime fly ash and control mortar bars have similar response indicating that high lime fly ash is ineffective in significantly mitigating the expansions to below those of control. This behavior is seen irrespective of the effect of the fly ash dosage on expansions.

The correlation between the fly ash dosage and the percent expansions shown in figure 4.78 indicate a similar trend as observed with the other three reactive aggregates (SP limestone, NM Rhyolite and NC argillite). Low lime and intermediate lime fly ash show a good correlation while high lime fly ash has a moderate correlation. However, with respect to high lime fly ash, compared to the poor to moderate correlation observed with other three

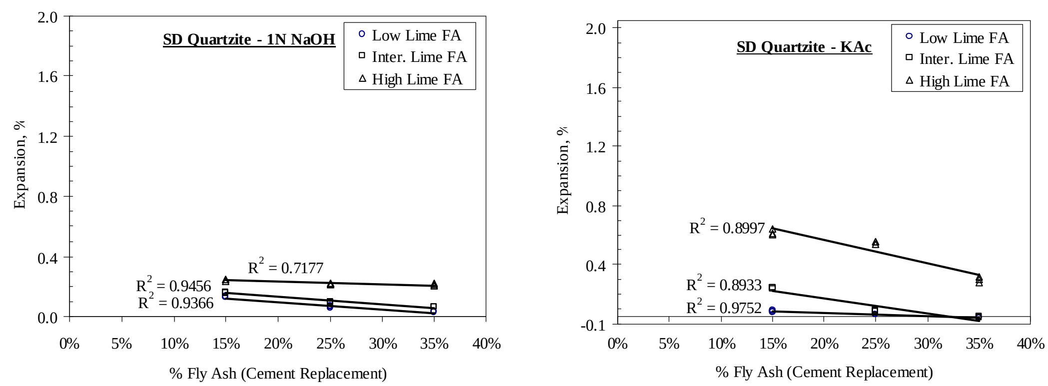



Figure 4.78 Correlations between the 14 Day Mortar bar Expansion, Fly Ash Dosage and Fly Ash Type in 1N NaOH and Potassium Acetate Exposure for SD Quartzite Containing Mortar Bars.

In summary, it can be said that the statistical difference between the three fly ash dosages in 1N NaOH exposure is aggregate specific. NM rhyolite and SD quartzite show that the difference between the expansions at 15%, 25%, 35% and control (0%) is statistically significant, while for SP limestone and NC argillite 15% and 25% dosage show similar effectiveness and hence their difference is not significant. In potassium acetate exposure, except for SD quartzite, the remaining three aggregates show that they have a statistically significant difference between the 35% dosage and control (0%) expansions irrespective of the fly ash type.

With regards to the correlation between fly ash dosage and percent expansion at 14 days as a factor of the type of fly ash used, low lime and intermediate lime fly ash show a good correlation while high lime fly ash show a poor to moderate correlation. This trend indicates that as the type of fly ash changes from high to intermediate to low lime, the mortar bar expansions reduce on increasing the dosage from 15% to 25% to 35% for all the three fly ash types. However, NM rhyolite mortar bars in potassium acetate exposure with high lime fly ash were an exception to this rule as it showed an increase in the expansion with an increase in the fly ash dosage.

#### 4.9.2 Influence of Lime Content of Fly Ashes on Expansions of Mortar Bar Tests

Based on the LSD grouping of the expansions of the Spratt limestone mortar bars made with fifteen fly ashes with varying lime contents (%CaO varying from 1.27% to 29.85%) at 25% dosage. in 1N NaOH exposure, it can be observed that the low lime and the intermediate lime fly ashes, in general, have similar difference between them and hence their difference is not statistically significant (refer table F.5 in Appendix). Similarly, high lime fly ashes and control have similar difference between them and hence are not statistically significant. Of all the low lime fly ashes in 1N NaOH exposure, LL3 fly ash had a similar response like four of the six intermediate lime fly ashes (IL1 to IL4) and one of the low lime fly ashes (LL2). This indicates that the effectiveness of these low and intermediate lime fly ashes was similar at 25% dosage. Two of the four high lime fly ashes (HL3 and HL4), having the highest %CaO contents among the fifteen fly ashes, have a similar response when compared to control mortar bar expansions. This indicates that at 25% dosage high lime fly ash is not effective in significantly reducing the expansions when compared to control.

In potassium acetate exposure, the similarity in the difference between the low and intermediate lime fly ashes is more pronounced compared to that in 1N NaOH (refer table

F.5 in Appendix). With the exception of intermediate lime fly ash (IL5), the other five intermediate lime fly ashes show similar effectiveness as the four low lime fly ashes. These results corroborate the 14 day expansion results presented in figure 4.4, where a sudden increase in the expansion of the mortar bars containing fly ashes with lime content greater than 15% ( >52% for CaO of cement fly ash blend) was observed. On the contrary, the high lime fly ashes (HL1 to HL4) show a significant statistical difference between each other and with the other low and intermediate lime fly ashes too. The only exception to this statement were the high lime fly ashes HL3 and HL4 that had an insignificant difference between them, but their expansions were significantly higher than 0.1% at 14 days

### 4.9.3 Influence of Dosage of Slag and Aggregate Type on Expansions of Mortar Bar Tests

The results of the LSD test (table F.6 in Appendix) indicate that 40% and 50% dosage of slag have a similar influence in mitigating the expansions in 1N NaOH exposure and their difference is not significant. While in potassium acetate exposure, the difference between the two dosages is statistically significant. This is also seen in figure 4.31 where the expansions of mortar bars containing 40% slag are much higher compared to those containing 50% slag when exposed to potassium acetate. However, irrespective of the exposure condition, the difference between any of the two slag dosages and control expansions is statistically significant irrespective of the type of aggregate used.

LSD results also indicate that in 1N NaOH exposure, with the exception of NM rhyolite, the remaining four aggregates (SP, NC, SD and IL) show similar difference between them irrespective of the dosage of slag used and hence the differences between them are statistically not significant. The high expansions of NM rhyolite make it significantly different from the other aggregates in both 1N NaOH and potassium acetate. NC argillite has similar differences in expansions with other four aggregates in both the solutions making their differences statistically not significant. SD quartzite has insignificant difference between the expansions with NC argillite and SP limestone, but is significantly different from IL dolomite in potassium acetate.

The linear regression analysis of the 14 day expansion results of all the five aggregates at 40% and 50% slag dosages in 1N NaOH and potassium acetate is shown in figure 4.79. In 1N NaOH, it is evident that there is a good inverse correlation between the 14 day expansions and the dosage of slag for all the five aggregates. This indicates that as the dosage of slag increases from 0% to 50%, the expansions reduce significantly. However, in potassium acetate exposure only three of the five aggregates (SP, NC and IL) show a good correlation between the expansions and the slag dosage, while NM rhyolite ad SD quartzite show moderate and poor correlations respectively. This variability is due to the sudden reduction in expansion of the mortar bars on increasing the dosage from 40% to 50% and for both NM rhyolite and SD quartzite aggregates.

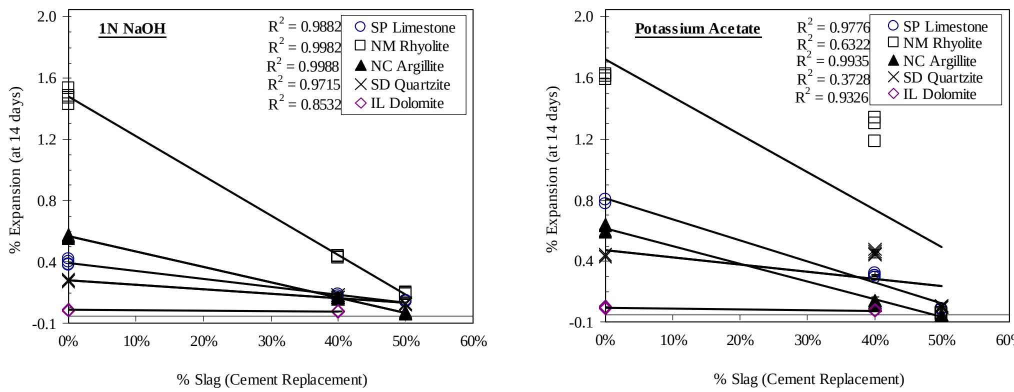



Figure 4.79 Correlations between the 14 Day Mortar bar Expansion and Slag Dosage in 1N NaOH and Potassium Acetate Exposure for Mortar bars Containing SP, NM, NC, SD and IL aggregates.

## 4.9.4 Influence of Fly Ash Dosage and Fly Ash Type on Expansions of Concrete Prism Tests

The LSD results of the of concrete prism expansions at 365 days test age (refer table F.7 in Appendix) as a factor of fly ash dosage and the type of fly ash for Spratt limestone indicate that the difference between the expansions of concrete prisms, irrespective of the type of fly ash used, at 0%, 25% and 35% dosage are significant.

Contrary to this, except for the difference between 0% and 35% fly ash dosage, the difference between 0% and 25% and 25% and 35% are statistically not significant. Analyzing the LSD results from the perspective of fly ash type, irrespective of the fly ash dosage used, it indicates that the differences between the expansions of the concrete prisms containing the three types of fly ash and control are significant in 1N NaOH. In potassium acetate exposure, with the exception of difference between low lime fly ash (LL) and intermediate lime fly ash (IL), the differences between the three fly ashes and control are significant. This indicates that low lime at intermediate lime fly ash have a similar effect in mitigating the expansions in potassium acetate.

For New Mexico aggregate, 25% and 35% dosage have similar difference in concrete prism expansions in 1N NaOH while in potassium acetate exposure 0% and 35% dosage have a similar difference irrespective of the fly ash dosage. This indicates that 25% fly ash aggravates the expansions and hence the difference between 25% dosage, 0% and 35% are significant. However, with regards to the fly ash type, in 1N NaOH NM aggregate has the same LSD results as obtained for Spratt aggregate tests. In potassium acetate, high lime fly ash (HL) has high expansions and hence its difference in expansions with the control and other two fly ashes (IL and LL) is significant. The similar difference between the low lime fly ash (LL), intermediate lime (IL) fly ash and control indicates that, irrespective of the dosage at which these fly ashes are used, using these fly ashes does not provide any significant reduction in the expansions compared to control.

### 4.9.5 Influence of Dosage of Slag on Expansions of Concrete Prism Tests

The LSD test results of the concrete prism expansions in 1N NaOH with 40% and 50% dosage of slag show similar difference between them, but are significantly different compared to the control (refer table F.8 in Appendix). This indicates that the use of slag at both these dosages, irrespective of the aggregate used, is effective in reducing the expansions. However, this does not mean that slag reduces the expansions to below 0.04% acceptance limit for ASTM C 1293 test.

In potassium acetate the differences between control, 40% and 50% dosage are all similar indicating that slag is not effective in reducing the expansions at any of the two dosages irrespective of the aggregate type used.

Analyzing the LSD results from the perspective of the type of aggregate used, it indicates that in 1N NaOH Spratt and New Mexico and Spratt and Illinois dolomite (IL) aggregate samples have similar differences between them and are not statistically significant. However, the difference between IL and NM is significant and it shows that IL has low expansions being a non-reactive aggregate and NM has high expansions being a reactive aggregate.

In potassium acetate, the difference in concrete prism expansions between all the three aggregates is significant. This indicates that IL has low expansions, SP has moderate expansions and NM has high expansions making the differences between the them high and hence statistically significant.

## 4.10 pH Studies

This section presents the results and discussion of the pH studies conducted on the interaction of cement-fly ash, cement-slag and control cement paste samples with 1N NaOH and potassium acetate solution. In addition to the cement paste samples, the pH of the soak solutions were measured at the end of the mortar bar tests (Standard and Modified ASTM C 1567 tests) at 28 days and, at 180 days and 365 days in the concrete prism test (Modified ASTM C 1293 test).

### 4.10.1 Cement Paste Samples

Figure 4.80 presents the change in the pH over the period of soaking the cement paste samples in 1N NaOH and potassium acetate. The influence of fly ash dosage of the three fly ashes on the pH of the soak solutions is also shown in this figure. These results are compared with the control cement paste results containing no fly ash.

Based on the pH measurements it is evident that the behavior of 1N NaOH and potassium acetate solutions is different from each other when exposed to cement paste samples with or without any fly ash. The pH of 1N NaOH was measured to be 13.7 at the start of the test and was 10.8 for potassium acetate. The pH of 1N NaOH was almost the same (13.66) at the end of the test for the control samples. On the contrary, the pH of the potassium acetate solution jumped by almost three orders (13.8) just after 3 days of soaking the cement paste samples in it. However, following the initial jump the pH of the solution remained stable till the end of the test (13.83 at 21 days).

This contrasting behavior in the pH measurements of the two solutions was evident even for solutions exposed to cement-fly ash paste samples. With regards to the type of fly ash used in the cement paste samples, a trend was seen in the pH change of both the soak solutions. In 1N NaOH, it was observed that irrespective of the fly ash dosage used in the cement paste, the type of fly ash used had a negligible influence on the pH of the solution. However, for all the three fly ashes, the pH of the 1N NaOH soaking solution was lower than the initial pH at the end of the test. The increasing dosage of the fly ash did not show any conclusive increasing or decreasing trend in the pH values.

In potassium acetate, the type of fly ash and dosage did have an influence on the pH of the solution in the early age of the test. However, none of the three fly ash types at any of the three dosages (15%, 25% and 35%) could suppress the pH of the soak solutions to lower than or equal to its initial pH. The jump in the pH at the early age (3 day) of exposure was similar to what was observed in the soak solution exposed to control cement paste samples. However, it was evident that low and intermediate lime fly ash samples lowered the pH of potassium acetate more than the high lime fly ash samples. On increasing the dosage of fly ash in the cement paste samples from 15% to 35%, it was seen that fly ash at 15% increased the pH of potassium acetate soak solution to even higher than the control soak solutions. At the 3 day pH measurement, it appeared that 25% and 35% fly ash dosage is able to suppress the pH of the solution less than control but, comparing the 21 day pH values of potassium acetate solution exposed to control and 25% or 35% fly ash samples, there is a negligible difference in the pH values.

This pH mechanism supports the expansions trend of the mortar and concrete prisms discussed earlier in this chapter.

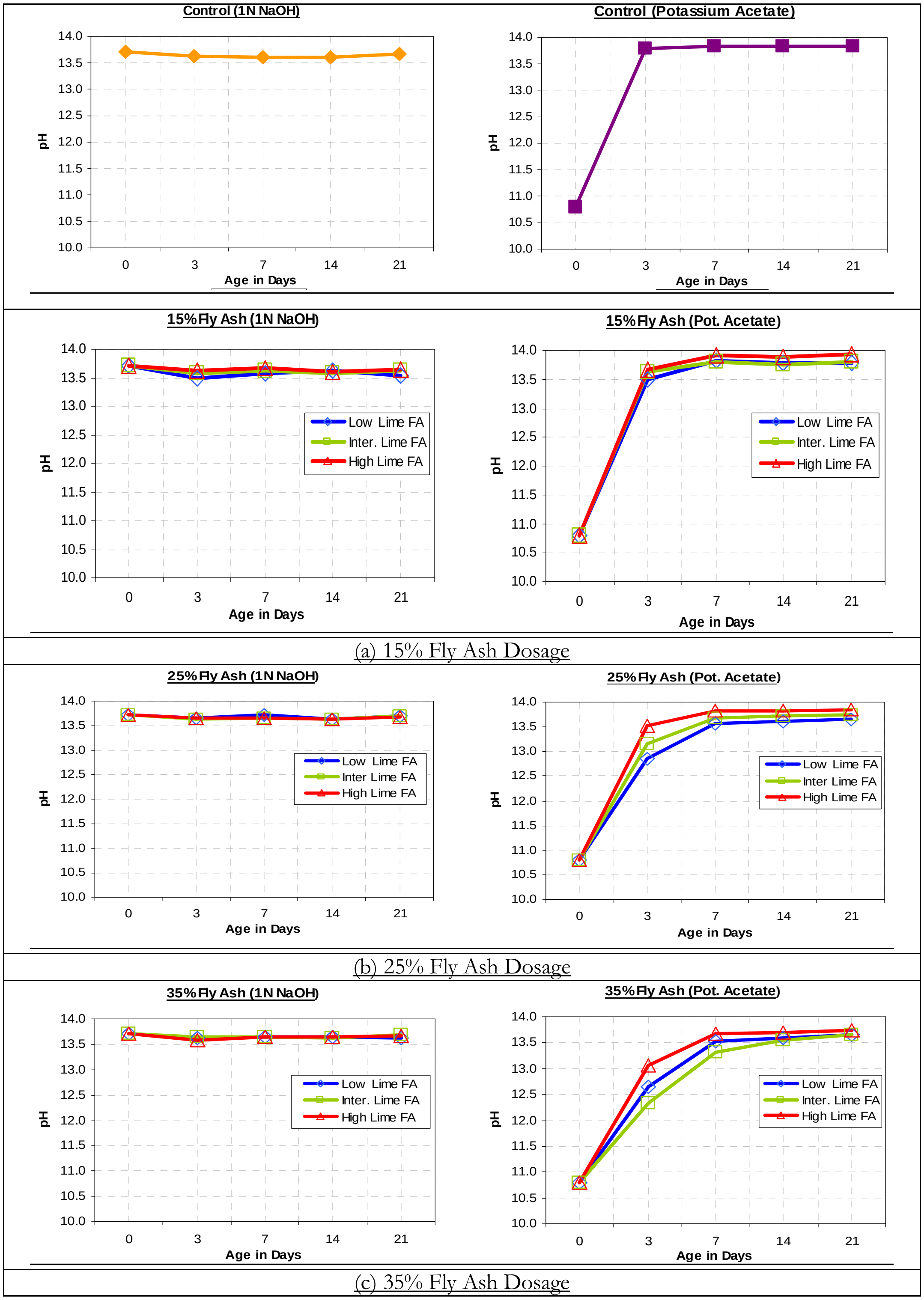


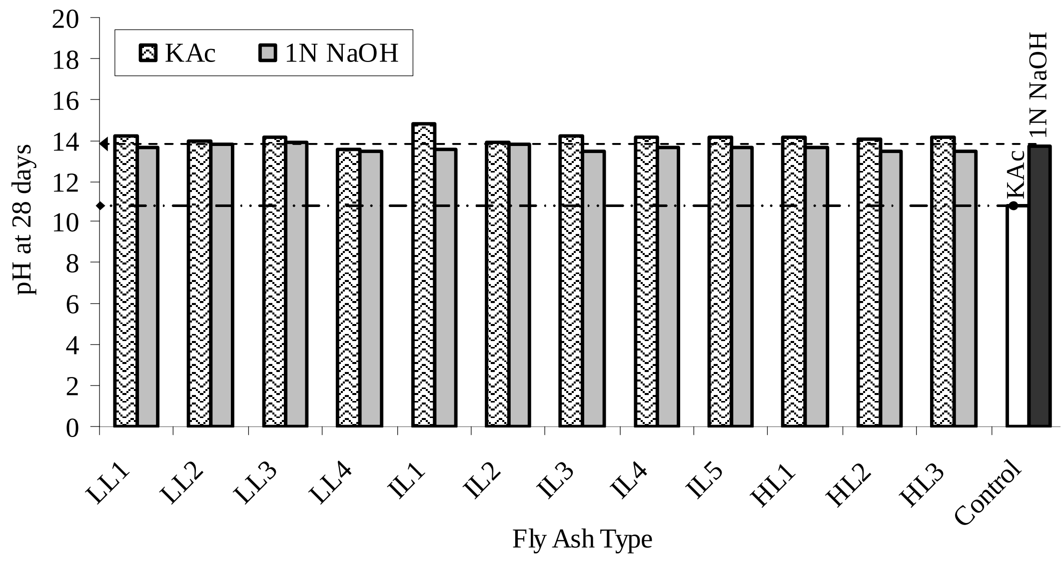

Figure 4.80 pH Measurements of Soak Solutions Exposed to Mortar Bars Containing Spratt Aggregate and Fly Ash at Dosages of 0%, 15%, 25% and 35%.

## 4.10.2 pH of Soak Solutions Exposed to Mortar Bars

Figure 4.81 shows the 28 day pH measurements of two soak solutions- 1N NaOH and potassium acetate (KAc), exposed to mortar bars containing fly ash, based on their lime (%CaO) content, at 25% dosage in combination with Spratt aggregate. These are compared with the 'Control' pH measurements of the soak solutions that were not exposed to mortar bars.

The pH results of 1N NaOH and KAc show a similar trend to what was observed in the solutions exposed to cement paste samples. Here too the pH of 1N NaOH solution after 28 days of exposure to mortar bars is almost the same as that of control. In KAc, the pH had increased to 14.0 or more for almost all of the mortar bar samples. This jump in the pH from supports the results of the cement paste studies.

Another observation that can be noted from these pH results is that the type of fly ash used or the lime content (%CaO) of the fly ash did not have any influence on the pH of the soak solutions. This is justified by conducting a regression analysis of the results of the 28 day pH measurements of the soak solutions in which the mortar bars were soaked and, the length change expansions at the same age of those mortar bars. The correlation shown in figure 4.82 concludes that there is a poor relation between the pH of 1N NaOH and potassium acetate soak solution and the expansions of the mortar bars and that the addition of any class of fly ash at 25% dosage does not have any significant influence on lowering its pH.




*Note: 'Control' indicates pH of soak solutions before soaking the mortar bars in them, i.e. pH at the start of the test.*

Figure 4.81 pH Measurements of Soak Solutions Exposed to Mortar Bars Containing Three Classes of Fly Ashes at 25% Dosage in Combination with Spratt Aggregate after 28 Days Exposure

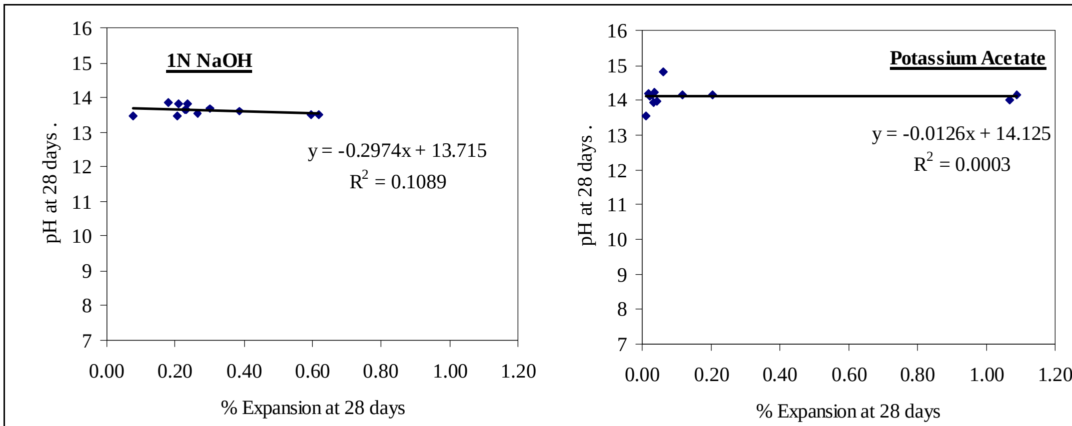



Figure 4.82 Correlation Between pH of Soak Solutions Exposed to Mortar Bars Containing Three Classes of Fly Ashes at 25% Dosage with Spratt Aggregate and Their Respective Expansions at 28 days Test Age.

### 4.10.3 pH of Soak Solutions Exposed to Concrete Prisms

Figure 4.83 shows the results of the pH measurements of soak solutions measured at 180 days and 365 days of exposure to concrete prisms in the modified ASTM C 1293 test. The concrete prisms were made with Spratt or New Mexico aggregate in combination with three fly ashes having a varied chemical composition, lime content in specific, at 25% and 35% dosage. The pH results are compared with the pH of the control soak solutions that are not exposed to concrete prisms.

Irrespective of the type of aggregate and the dosage of fly ash used, the pH of the soak solutions represent the same trend as observed for the soak solutions exposed to cement paste and mortar bar samples. The pH of 1N NaOH does not change significantly with respect to the control at both 180 and 365 days of soaking the concrete prisms. However, it is evident that for both the aggregates at both the dosages, the pH at 180 and 365 days is lower than the control (13.71). This indicates that the addition of fly ash did help in reducing the hydroxyl ion concentration by a small amount. For the potassium acetate soak solution, the pH at 180 days is higher by almost three orders in reference to the control (10.8) and it has a negligible increase/degrease when measured after 365 days of soaking of the concrete prisms. The type of fly ash used in the concrete prisms does not seem to have a significant influence on the pH of the soak solutions. However, in all the potassium acetate solutions the pH is always higher than 14.0 at 180 days and it stays above 14.0 even at 365 days. This highly alkaline environment is conducive for ASR to propagate.

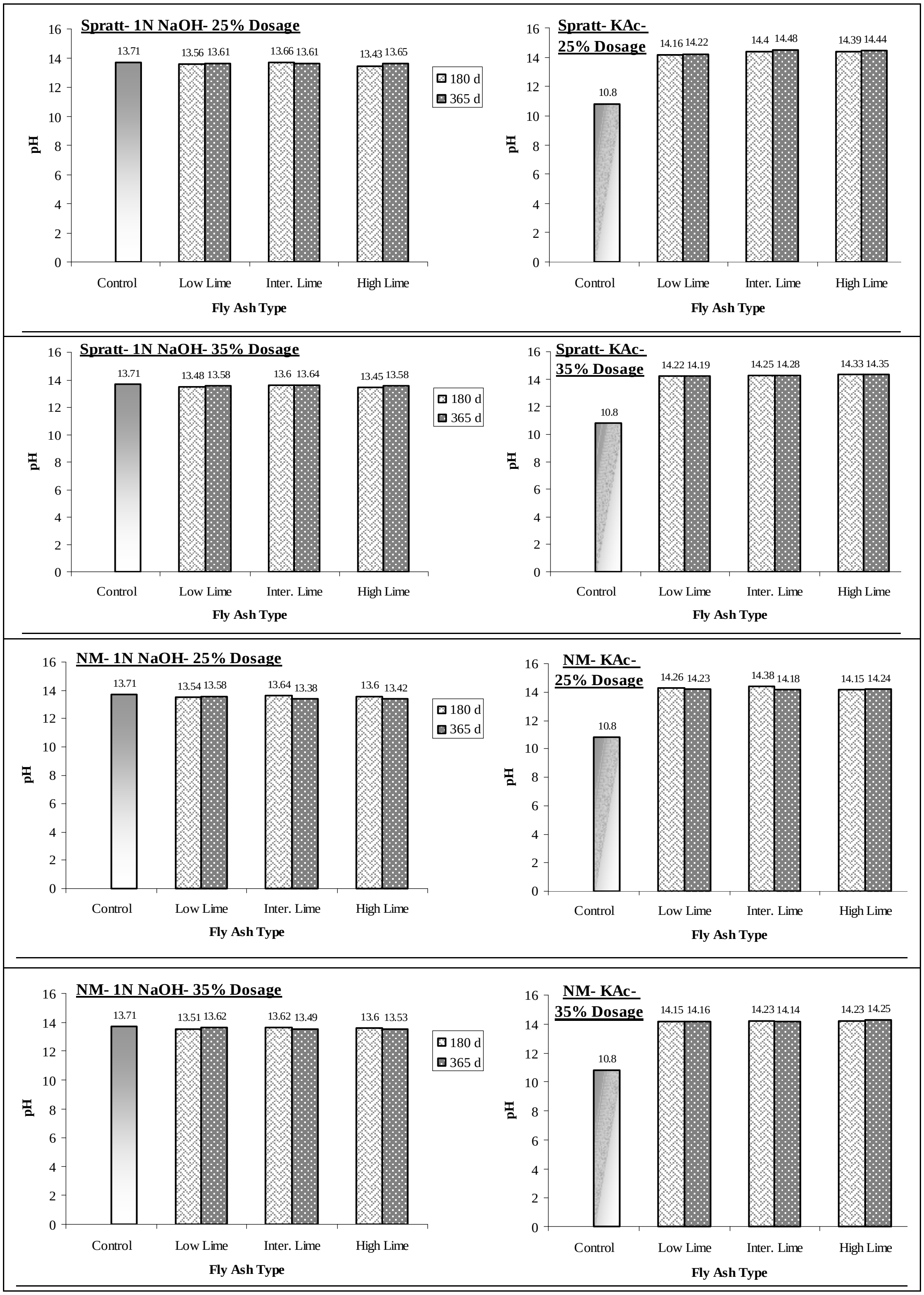



Figure 4.83 pH Measurements of Soak Solutions of ASTM C 1293 (Modified) Tests Containing Spratt and NM Rhyolite Aggregate with Fly Ash at 180 and 365 Days Test Age.

Figure 4.84shows the pH measurements of soak solutions measured at 180 days and 365 days of exposure to concrete prisms in the modified ASTM C 1293 test. The concrete prisms were made with Spratt, New Mexico or Illinois aggregate in combination with slag at 40% and 50% dosage.

The results show the same trend as seen in figure 4.83 for concrete prisms made with fly ash. However, it should be noted that the pH of the potassium acetate soak solution increased to above 14.0 inspite of using a non-reactive Illinois dolomite aggregate in the concrete prisms. This indicates that the pH of the soak solution is a factor of the reaction between the cement paste (or cement-slag paste) and potassium acetate and the aggregate does not play a role in this. The dosage of slag did not have a significant influence on the pH of the soak solutions.

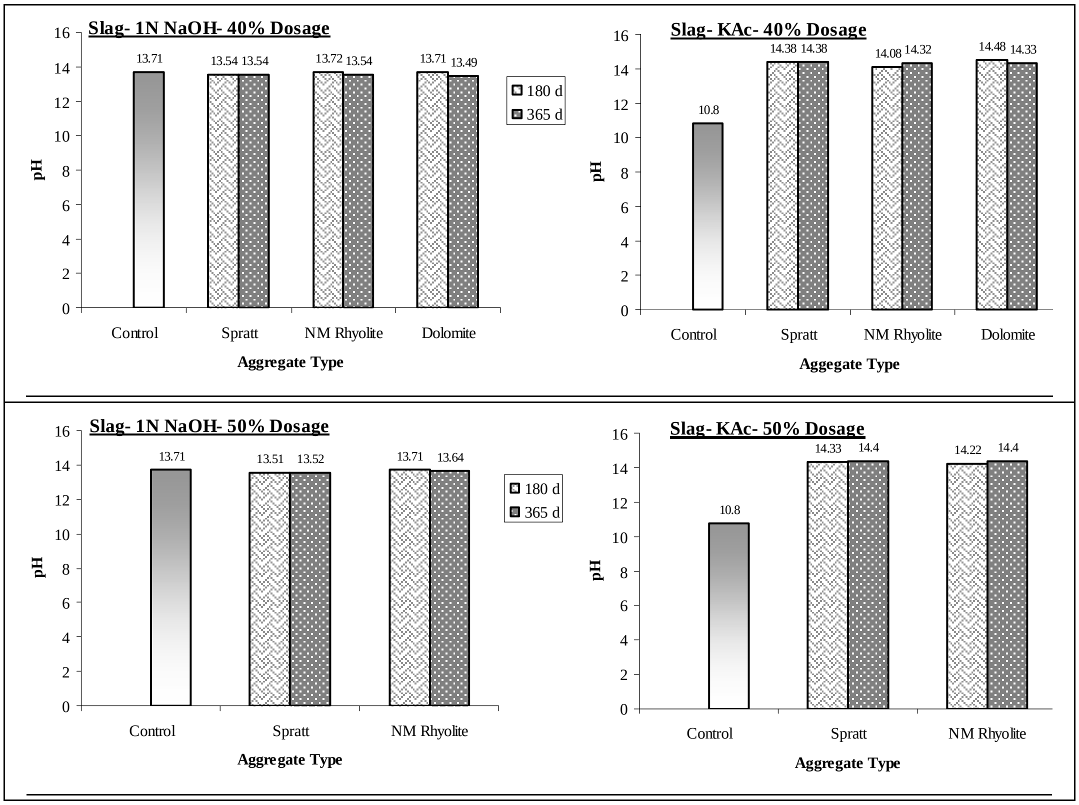



Figure 4.84 pH Measurements of Soak Solutions Exposed to Mortar Bars Containing Three Aggregates at 40% and 50% Dosage of Slag at 180 and 365 Days Test Age.

The indifference of the pH of 1N NaOH solution after an exposure to cement paste or mortar bar samples for 21 and 28 days respectively, is due to the equilibrium maintained between the pore solutions of the cement paste/mortar bar and the soak solution. A previous study (Rangaraju et al 2007) indicated that the pore solution extracted from the concrete prisms tested under standard ASTM C 1293 test has a concentration of about 1N, which is the same as that of the soak solution. This leads to an equilibrium state between the soak solution and the pore solution of the cement paste/mortar bar samples. However, the increased expansions over the test period in the mortar bar tests is due to the already high pH of 1N NaOH solution that exacerbates the ASR reaction by providing a source of OH- ions.

The sudden increase in the pH of the potassium acetate soak solution exposed to cement paste and mortar bar samples supports the findings of Stark et al. (Stark et al. 2006). The formation of portlandite-$Ca(OH)_2$, due to the hydration of cement paste and its subsequent contact with the potassium acetate soak solution leads to the formation of calcium acetate leading to a drop in the concentration of $Ca^{2+}$ ion which in turn leads to the dissolution of new portlandite into the solution to maintain an equilibrium state. The dissolution of portlandite releases new calcium ($Ca^{2+}$) and hydroxide ions ($OH^{-}$) leading to an increase in the pH of the solution (eq. 1). However, as the pH increases the solubility of portlandite decreases and the pH increase is possible only as long as there is an availability of free $Ca^{2+}$ ions in the solution and they can come in contact with the acetate ions.

$$\mathbf{2K^{+} + 2CH_3COO^{-} + Ca^{2+} + 2\,OH^{-} \leftrightarrow 2Ca(CH_3COO)_2(aq.) + 2K^{+} + 2OH^{-}} \quad \textit{(eq. 1)}$$

## 4.11 Silica Dissolution Study- Inductively Coupled Plasma (ICP) Test

This section presents the results of the Inductively Coupled Plasma (ICP) test conducted on filtered 1N NaOH and potassium acetate solutions to determine the concentration of silicon, calcium, sodium and potassium in them. Fused silica particles were soaked in these solutions with or without calcium hydroxide (CH) added to them for up to 672 hours (28 days). One set of these solutions was stored at room temperature and the other at $38^{0}C$. The solutions were filtered at 8.5, 26, 48, 168, 384, 504 and 672 hours and the filtered solutions were analyzed by ICP for their elemental composition.

Figure 4.85 shows the results of the ICP test and the silicon concentration detected in 1N NaOH and potassium acetate at different storage intervals (in hours). It also shows the influence of temperature and presence of calcium hydroxide on the silica dissolution potential. Figure 4.86 shows the concentration of four elements in the filtered solutions of 1N NaOH and KAc with or without CH in them.

Results shown in figure 4.85 indicate that at room temperature the dissolution of silica is higher in 1N NaOH solution (1534 ppm) compared to that in potassium acetate solution (163 ppm). The dissolution of silica in the presence of calcium hydroxide (CH) was lower in both 1N NaOH and KAc compared to that without CH. On storing the solutions at $38^0$C, the dissolution of silica increased by about 4 times in both plain 1N NaOH (6884 ppm) and in 1N NaOH with CH (2379ppm). However, in potassium acetate there was a negligible influence of temperature on the silica dissolution with or without CH in it.

Figure 4.86 shows the changes in sodium, potassium and calcium ion concentration along with the silicon ion concentration in 1N NaOH and KAc solutions with or without CH. It is obvious that in 1N NaOH solution the concentration of sodium ions will be the highest and ideally there will be no potassium or calcium present in it. Whatever potassium or calcium is detected may be due to the impurities present in the solution. In plain 1N NaOH, the increase in the silica concentration over time does not lead to any significant increase or decrease in the concentrations of other three elements. However, in the presence of CH, as the silica concentration increases the sodium concentration decreases while the calcium and potassium ion concentrations do not have any significant changes.

In potassium acetate, the dissolution of silica was negligible in comparison to that in 1N NaOH. However, the presence of calcium hydroxide in potassium acetate suppressed the silica dissolution and a lower concentration (23 ppm) was detected in comparison to that detected in plain potassium acetate (163 ppm). However, there was an increase in the calcium concentration where the concentration increased fro 278ppm (at 8.5 hours) to 1001ppm (at 672 hours).

## SEM Analysis

The SEM analysis was conducted on fused silica particles exposed to 1N NaOH and potassium acetate deicer solution with and without the presence of lime (CH) in them for 90 days. The silica particles were washed with water over a 150 micron sieve, followed by drying them for a day at 38$^{o}$C and embedding them in epoxy. The epoxy embedded samples were polished the same was as described in section 3.3.8.

Figure 4.87 shows the SEM images of fused silica particles exposed to 1N NaOH without lime addition and with it. It is evident from these figures that the silica particles have reacted as seen from the edges of the particles. Comparing the silica particles exposed to plain 1N NaOH and with lime added to the solution, it is evident that there are more reacted articles in the plain 1N NaOH than in 1N NaOH with lime (CH).

Figure 4.88 shows the SEM images of fused silica particles exposed to 1N NaOH without lime addition and with it Similar to the reacted particles observed in 1N NaOH solution, the particles exposed to potassium acetate too had reacted edges. The amount of reacted articles exposed to plain potassium acetate solution and with lime in it was almost similar.

However, comparing the reacted particles in 1N NaOH to those exposed t potassium acetate, there was much more reacted silica in 1N NaOH. This is consistent with the silica concentration obtained in the ICP test. The SEM images could not provide conclusive evidence with regards to the role of lime in suppressing the silica dissolution when exposed to 1N NaOH and potassium acetate.

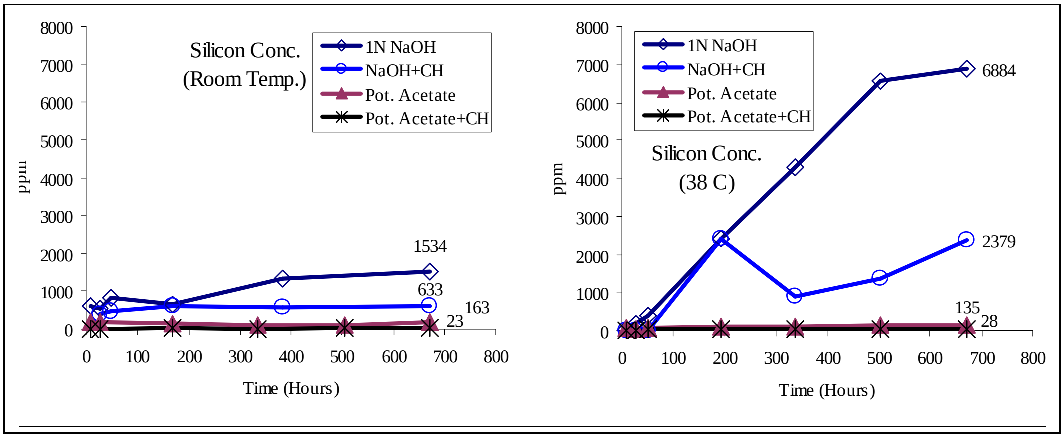



Figure 4.85Concentration of Silicon Detected in 1N NaOH and KAc in ICP Test

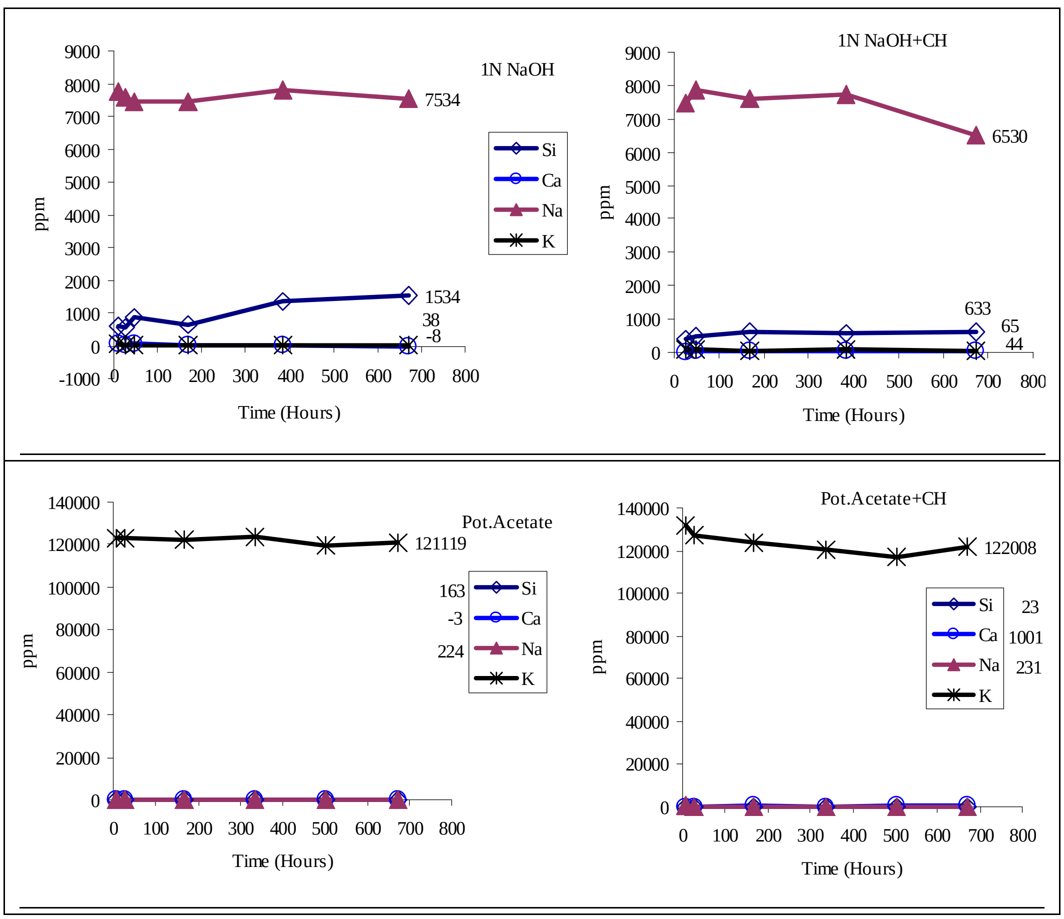



Figure 4.86 Concentrations of Selected Elements in 1N NaOH and KAc in ICP Test

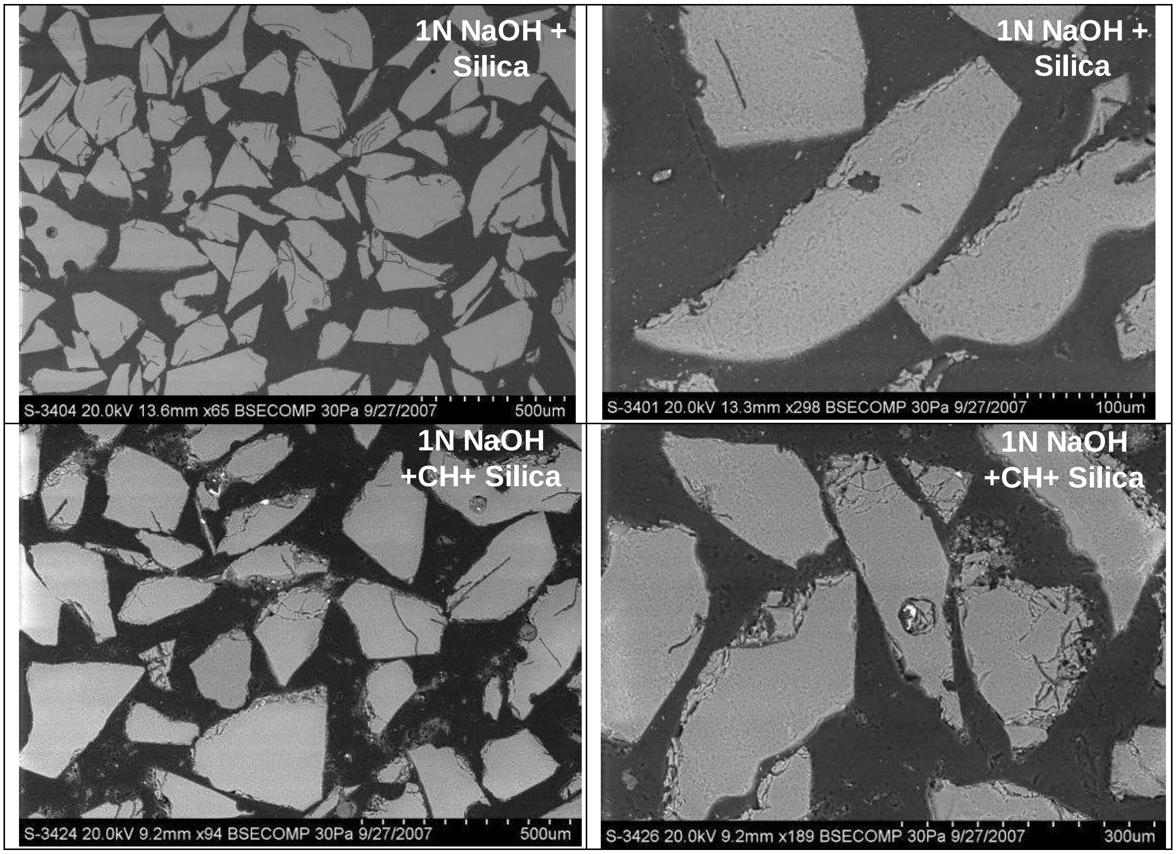



Figure 4.87 SEM Images of Fused Silica Particles Exposed to 1N NaOH With and Without the Presence of Calcium Hydroxide for 90 Days

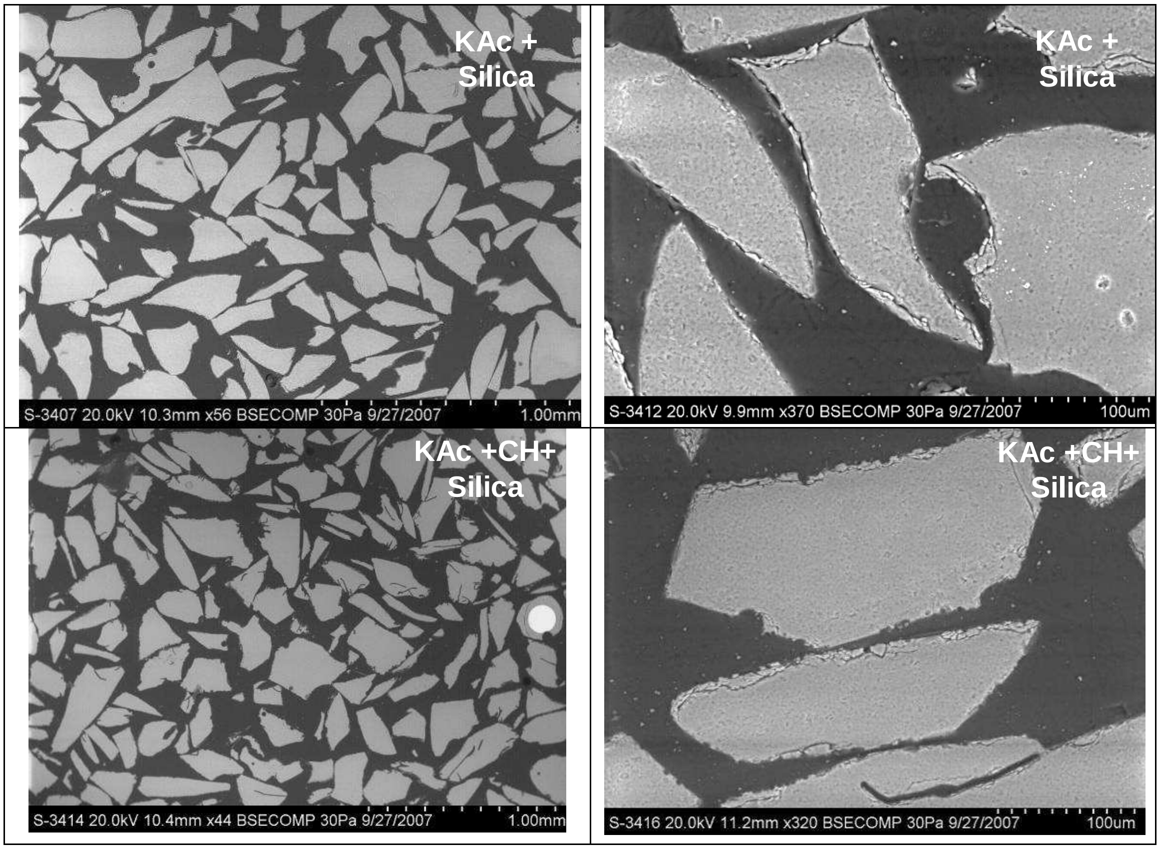



Figure 4.88 SEM Images of Fused Silica Particles Exposed to Potassium Acetate With and Without the Presence of Calcium Hydroxide for 90 Days

## 4.12 Summary

This section is aimed summarizing the results and discussion of all the tests discussed in the previous sections of this chapter and draw out the principal findings of this study.

### Evaluation of Fly Ash in the Standard and Modified ASTM C 1260 and 1567 Tests

- Of the five aggregates tested, New Mexico rhyolite had the highest expansions followed by Spratt limestone, North Carolina argillite, South Dakota quartzite and Illinois dolomite.
- Irrespective of the type of fly ash used, 35% fly ash dosage provided the highest mitigation of the expansions of the mortar bars in both standard (i.e. in 1N NaOH solution) and modified (i.e. in potassium acetate deicer solution exposure) ASTM C 1567 tests. This was true for all the four reactive aggregates as confirmed by the statistical analyses that indicated the differences between the 14 day expansions of the mortar bars containing 15% and 25% and were significantly different from those containing 35% dosage.
- Of the three categories of fly ashes tested, low lime fly ash provided the most mitigation in both 1N NaOH and potassium acetate exposure conditions. Low lime fly ash was most effective at 35% dosage for all the four reactive aggregates by controlling the expansions to below 0.1% at both 14 and 28 days. However, even a dosage of 25% was adequate to control the expansions to 0.1% for three of the four reactive aggregates (SP, NC and SD) in both 1N NaOH and potassium acetate. A 15% dosage of low lime fly ash was effective only for NC argillite and SD quartzite only in potassium acetate exposure.

- Similar to the low lime fly ash, intermediate lime fly ash was effective at 25% and 35% dosage for three of the four reactive aggregates except NM rhyolite in potassium acetate exposure. However, only 35% dosage provided adequate mitigation in both 1N NaOH and potassium acetate for all reactive aggregates tested except for NM rhyolite.
- High lime fly ash was found to be ineffective in controlling the expansions to below 0.1% at 14 days for all the four reactive aggregates in both the standard and modified 1567 tests. In fact, at 15% and 25% dosage the expansions of mortar bars soaked in 1N NaOH were similar to those observed in control mortar bars. Mortar bars with high lime fly ash soaked in potassium acetate showed higher expansions than the control at 15% and 25% dosage for three of the four reactive aggregates except for SP limestone.
- The deleterious interactions between high lime fly ash and potassium acetate can be seen even in mortar bars containing non-reactive IL dolomite aggregate, where the mortar bar expansions in the presence of potassium acetate were higher than those observed in control mortar bars without any fly ash.

  This suggests that though the reactivity of the aggregate plays an important role in the overall expansion, potassium acetate has the potential to react with the fly ash in the cement paste and create deleterious expansions.
- SEM analysis of mortar bar samples subjected to the standard and modified ASTM C 1567 tests were conducted for SP limestone and 25% fly ash containing mortar bars. Certain distinguishing features were observed during the SEM analysis of the samples exposed to 1N NaOH and potassium acetate.

Spratt limestone mortar bars with 25% dosage of low and intermediate lime fly ash showed moderate levels of cracking in the cement paste and aggregate-paste interface regions in both 1N NaOH and KAc. However, the paste was infused with sodium in 1N NaOH exposure samples and, with potassium in KAc exposed samples. The cracking was higher in 25% intermediate lime fly ash samples exposed to KAc when compared to the 1N NaOH exposed samples.

High lime fly ash containing mortar bar samples showed extensive cracking in the cement paste and through the aggregate particles in both the exposure conditions. However, the cracks within the aggregate particles were wider in KAc exposed samples than those exposed to 1N NaOH and this was consistent with the high levels of expansions observed in the modified ASTM C 1567 test. Dense ASR gel rim formation was observed in KAc exposed samples. In 1N NaOH samples, the walls of the cracks within the aggregate were lined with ASR gel, while in KAc most of the ASR gel was found at the aggregate paste interface of the cracked aggregate. Presumably, the ASR gel might have migrated from the cracks into the paste.

Influence of Chemical Composition of Fly Ashes on the Mortar Bar Expansions

- The correlations between the individual chemical constituents of fifteen different fly ashes and the 14 day expansions of the mortar bars containing these fly ashes at 25% dosage with SP limestone aggregate provide useful information in characterizing the fly ashes for their ASR mitigation potential.

- Lime content (%CaO) and sulfate content (%$SO_3$) of the fly ash were found to have a strong positive correlation with the 14 day expansions of the mortar bars in the standard and modified ASTM C 1567 tests.
- The $\mathbf{CaO_{eq}/\ SiO_{2\ eq}}$ ratio and the $\mathbf{CaO/(SiO_2)^2}$ provide a strong positive correlation with the 14 day expansions in the standard and modified ASTM C 1567 tests and can be used in selecting fly ashes based on chemical composition.

Evaluation of Fly Ash in the Modified ASTM C 1293 Tests

The modified ASTM C 1293 concrete prism tests were conducted with two reactive aggregates (NM rhyolite and SP limestone) in combination with three fly ashes–low, intermediate and high lime, at 25% and 35% dosage. The trends in the concrete prism expansion results at the end of one year of testing were found to be consistent with those observed in the mortar bar test-ASTM C 1567 (standard and modified) at 14 days.

However, from a pass/fail criterion based on the expansion limits of the standard tests, there appears to be some inconsistencies. For example, intermediate lime fly ash at 25% dosage with SP limestone and, low lime fly ash at 25% and 35% with NM rhyolite in both 1N NaOH and KAc exposure, showed mixed response in the ASTM C 1567 and ASTM C1293 tests. The inconsistency in the results of the two tests might be due to the differences in the specimen size and the storage temperature of both the tests.

Evaluation Slag in the Standard and Modified ASTM C 1567 Tests

Slag provided adequate mitigation to control the mortar bar expansions to below 0.1% at 14 and 28 days in both 1N NaOH and KAc exposure at 50% dosage. This dosage was adequate for all the four reactive aggregates in KAc and 1N NaOH exposure but, in 1N

NaOH exposure NM rhyolite was the exception. 40% dosage was effective only with NC argillite aggregate in both the exposure conditions.

## Evaluation of Slag in the Modified ASTM C 1293 Tests

The effectiveness of slag at in the concrete prism test was similar to what was observed in the mortar bar tests. 40% dosage proved to be ineffective for both the aggregates tested (NM rhyolite and SP limestone) in both the modified C 1293 tests (1N NaOH and KAc exposure). However, at 50% dosage the results were conflicting as the concrete prism expansions exceeded the 0.04% acceptance limit of the C 1293 test in both 1N NaOH and KAc, whereas this dosage was adequate in controlling the expansions to below 0.1% in the mortar bar tests-ASTM C 1567.

## DME Measurements

DME values provide a useful tool in monitoring the physical integrity of the mortar and concrete samples over the length of the test. Irrespective of the exposure condition (1N NaOH or potassium acetate) there exists an inverse relationship between the expansions of the mortar bar containing reactive aggregates and concrete prism and their respective DME. It was clearly evident from the DME results that as the expansions increased the DME decreased indicating the deterioration of the mortar and concrete samples and subsequent loss of physical integrity. This observation was common for both fly ash and slag mortars.

## pH Measurements

pH of soak solutions -1N NaOH and potassium acetate deicer solution, exposed to cement paste vials, cement-fly ash paste, cement-slag paste, mortar bars and concrete prisms were compared to the solutions that were not exposed.

- With 1N NaOH solution, no significant changes in the pH occurred upon exposure to cement paste or cement-fly ash paste. The fly ash composition or the dosage dif not have a specific influence on the pH of the 1N NaOH soak solution.
- In potassium acetate deicer solution, it was observed that the type of fly ash and its dosage level had an influence on the pH of the solution at early age of the test (3 days and 7 days). However, irrespective of the type or dosage of the fly ash or slag used, pH at later ages was similar to each other (i.e. slightly in excess of 14).

## Discussion

It is believed that the sudden jump in the pH is due the formation of calcium acetate in the potassium acetate solution that leads to a drop in the calcium ion concentration and hence, more dissolution of portlandite from the cement paste to maintain equilibrium of calcium ions. Dissolution of portlandite releases hydroxyl ions into the solution leading to an increase in the pH.

In all the mortar bar and concrete prism tests with Illinois dolomite aggregate, the expansions observed were within the acceptable limits (below 0.1% at 14 days in ASTM C 1260 and C 1567 and below 0.04% in C 1293) with or without the use of fly ash or slag in both the exposure conditions. This confirmed the non-reactive nature of this aggregate and also confirmed that for an ASR to occur in the presence of potassium acetate, a reactive aggregate is essential in the mortar or concrete matrix.

This finding is corroborated by the findings in the SEM analyses of the mortar and concrete samples and also in the silica dissolution studies. The SEM analysis provided strong evidence suggesting that the distress in the form of cracking in most of the mortar and concrete samples was limited to the aggregate paste interface and the cement mortar matrix. In addition, all these samples had expansions that were high enough to exceed the acceptable limits. This observation is in somewhat of a contrast to what is typically observed in traditional ASR mechanism where the ASR gel forms within the aggregate and the expansive stresses generated cause the cracking of the aggregate and the mortar matrix.

This leads us to believe that the chemical interaction between potassium acetate and the aggregate particle preferentially occurs near the interfacial transition zone between the aggregate and the cement paste. However, the results of the silica dissolution study indicate that the dissolution of reactive silica is very low in presence of potassium acetate deicer in comparison to that found in 1N NaOH exposure. The silica dissolution is even lower when calcium hydroxide is present in the potassium acetate solution. Inspite of the low dissolution of silica in the presence of potassium acetate, a SEM analysis of the potassium acetate exposed fused silica particles provide evidence that the silica particle had reacted in the presence of potassium acetate.

These findings lead us to conclude that though the distress mechanism caused by potassium acetate involves the presence of alkali hydroxides and reactive silica, it is not similar to the traditional ASR in its reaction nature or dynamics.

# CHAPTER V CONCLUSIONS AND RECOMMENDATIONS

## 5.1 General

This chapter presents the conclusions of this study based on the results and discussion presented in chapter 4.. In addition some recommendations for further research and putting this research into practical use are also provided at the end of this chapter.

## 5.2 Conclusions

The following conclusions can be drawn from this study on the ASR mitigation potential of fly ash and slag in the presence of potassium acetate deicer

1. Potassium acetate deicer can cause deleterious reactions in Portland cement mortars and concrete containing reactive aggregates. No such reactions were evident in mixtures with non-reactive aggregate.
2. An overall ASR mitigating effect for most aggregates exposed to potassium acetate deicer and 1N NaOH was achieved by using intermediate or low lime fly ash at 25% or 35% cement replacement level.
3. High lime fly ash was ineffective at all the three replacement levels (15%, 25% and 35%) with all the reactive aggregates in both the standard (1N NaOH) and modified (KAc) tests. In fact, with two of the four reactive aggregates (NM rhyolite and SD quartzite) the expansions were higher than the control in presence of potassium acetate deicer solution.
4. The effectiveness of a particular fly ash at a particular replacement level was found to be dependant on the aggregate reactivity and chemical composition of the fly ash. In

general, except for mortar and concrete samples containing the highly reactive New Mexico rhyolite aggregate, low lime and intermediate lime fly ash was effective in mitigating the expansions at 25% and 35% dosage. The differences between the expansions of high lime fly ash and the intermediate and low lime fly ashes were significant in three of the four reactive aggregates tested in potassium acetate deicer exposure.

5. The lime (%CaO) content and the sulfate (%$SO_3$) content of the fly ash or cement/fly ash blend was found to have a significant influence on the mortar bar expansions. In general, an increase in the lime and sulfate content of fly ash was found to have a consequent increase in the mortar bar expansions.
6. The $CaO_{eq}$/ $SiO_{2\ eq}$ and the CaO/ $(SiO_2)^2$ ratios of the cement-fly ash blends provide a useful parameter in selecting fly ashes based on their chemical composition as an ASR mitigation alternative in potassium acetate deicer exposure conditions.
7. The expansion trends in the mortar bar and concrete prism tests were found to be similar. The correlations between the 14 day expansions in the standard and modified ASTM C 1567 were in general good with a few exceptions similarly, the correlation between the 365 days expansion results of the modified (type-1) and modified (type-2) were also found to be good.
8. Silica dissolution in potassium acetate solution was found to be lower than that observed in 1N NaOH solution. The silica dissolution in potassium acetate is further suppressed by the presence of calcium hydroxide. However, the low rate of silica dissolution in potassium acetate does not appear to prevent the reaction with the aggregates.

9. Slag was effective at 50% dosage in the standard and modified ASTM C 1567 mortar bar tests, but was found to be ineffective at this dosage in the modified ASTM C 1293 concrete prism test in the presence pf potassium acetate. A lower dosage of slag at 40% was found to be ineffective in both the mortar bar and concrete prism test.
10. The dynamic modulus of elasticity (DME) showed a good relationship to the expansions of the mortar bars and the concrete prisms and can provide a useful tool in monitoring the integrity of the samples under the standard and modified tests.

## 5.3 Recommendations

Based on the evaluation of mitigation potential of fly ashes and slag in the standard and modified ASTM C 1567 and C 1293 tests, the following recommendations are provided to generalize the findings from this study:

*Recommendations for Implementation*

1. Class F fly ash (low-intermediate lime fly ash) with a lime content less than 15% can be used at a minimum cement replacement level of 25% to significantly mitigate ASR effects of potassium acetate deicer solution.
2. For new concrete pavement construction to be exposed to potassium acetate deicer Grade 120 slag can be used at a minimum of 50% cement replacement level for mitigating ASR in presence of potassium acetate deicer solution.
3. Class C fly ash (with high levels of lime, i.e. >15%) should not be used when potassium acetate deicer application is anticipated on the concrete pavements.
4. The practicality of using high enough dosages of slag and fly ashes to mitigate ASR will depend on the particular construction application. Furthermore, the use of

ternary blends and other supplementary cementitious materials like silica fume and meta-kaolin needs to be explored for their effectiveness against ASR induced by pavement deicing chemicals such as potassium acetate.

### *Recommendations for Further Research*

1. The role of sulfate in the fly ash and cement in combination with their respective lime content and its relation to the expansions of the mortar and concrete samples in potassium acetate exposure should be studied in further detail.
2. The role of calcium acetate formation in the sudden increase in the pH of the potassium acetate deicer solution is not clearly understood and further studies are required.
3. Long term field studies should be conducted where concrete slabs or blocks made of mixtures with fly ash and slag are exposed to potassium acetate deicer in the same manner as applied in routine deicing applications. The samples should be monitored for their dimensional changes and chemical changes over a period of few winter seasons.

APPENDICES

# APPENDIX-A LENGTH CHANGE DATA FOR MORTAR BARS

Table A.1 Expansions (%) of Control Mortar Bars in Standard and Modified ASTM C 1260 test with Spratt Limestone and North Carolina Argillite (Sompura 2006)

| Spratt Limestone | | | | | | North Carolina | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Day | NaOH | | Day | Pot. Acetate | | Day | NaOH | | Pot. Acetate | |
| | Exp. (%) | Std. Dev. | | Exp. (%) | Std. Dev. | | Exp. (%) | Std. Dev. | Exp. (%) | Std. Dev. |
| 0 | 0.00 | 0.00 | 0 | 0.00 | 0.00 | 0 | 0.00 | 0.00 | 0.00 | 0.00 |
| 2 | 0.06 | 0.00 | 1 | 0.00 | 0.00 | 3 | 0.13 | 0.00 | 0.04 | 0.00 |
| 4 | 0.14 | 0.01 | 3 | 0.07 | 0.00 | 7 | 0.35 | 0.01 | 0.38 | 0.01 |
| 6 | 0.19 | 0.01 | 5 | 0.24 | 0.00 | 11 | 0.43 | 0.01 | 0.53 | 0.02 |
| 8 | 0.22 | 0.01 | 7 | 0.37 | 0.01 | 14 | 0.52 | 0.01 | 0.57 | 0.02 |
| 10 | 0.26 | 0.01 | 9 | 0.48 | 0.01 | 21 | 0.63 | 0.02 | 0.59 | 0.02 |
| 12 | 0.30 | 0.01 | 11 | 0.58 | 0.02 | 28 | 0.76 | 0.02 | 0.61 | 0.02 |
| 14 | 0.35 | 0.02 | 13 | 0.68 | 0.02 | | | | | |
| 16 | 0.40 | 0.02 | 14 | 0.74 | 0.02 | | | | | |
| 20 | 0.49 | 0.04 | 15 | 0.81 | 0.00 | | | | | |
| 24 | 0.64 | 0.02 | | | | | | | | |
| 28 | 0.76 | 0.00 | | | | | | | | |
| 32 | 0.87 | 0.00 | | | | | | | | |

Table A.2 Expansions (%) of Control Mortar Bars in Standard and Modified ASTM C 1260 test with South Dakota Quartzite and New Mexico Rhyolite (Sompura 2006)

| South Dakota | | | | | New Mexico | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Days | NaOH | | Pot. Acetate | | Days | NaOH | | Pot. Acetate | |
| | Exp. (%) | Std. Dev. | Exp. (%) | Std. Dev. | | Exp. (%) | Std. Dev. | Exp. (%) | Std. Dev. |
| 0 | 0.00 | 0.00 | 0.00 | 0.00 | 0 | 0.00 | 0.00 | 0.00 | 0.00 |
| 3 | 0.04 | 0.00 | 0.04 | 0.00 | 3 | 0.59 | 0.06 | 1.24 | 0.02 |
| 7 | 0.12 | 0.00 | 0.18 | 0.00 | 7 | 0.98 | 0.03 | 1.55 | 0.02 |
| 11 | 0.17 | 0.00 | 0.30 | 0.00 | 11 | 1.21 | 0.04 | 1.56 | 0.02 |
| 14 | 0.23 | 0.01 | 0.38 | 0.01 | 14 | 1.43 | 0.04 | 1.56 | 0.02 |
| 21 | 0.33 | 0.01 | 0.42 | 0.01 | 21 | 1.65 | 0.05 | 1.58 | 0.02 |
| 28 | 0.41 | 0.01 | 0.44 | 0.01 | 28 | 1.80 | 0.05 | 1.59 | 0.02 |

Table A.3 Expansions (%) of Control Mortar Bars in Standard and Modified ASTM C 1260 test with Illinois Dolomite

| Illinois Dolomite | | | | |
|---|---|---|---|---|
| Days | NaOH | | Pot. Acetate | |
| | Exp. (%) | Std. Dev. | Exp. (%) | Std. Dev. |
| 0 | 0.00 | 0.00 | 0.00 | 0.00 |
| 3 | 0.01 | 0.00 | 0.03 | 0.00 |
| 7 | 0.02 | 0.00 | 0.04 | 0.01 |
| 11 | 0.03 | 0.00 | 0.04 | 0.01 |
| 14 | 0.04 | 0.00 | 0.05 | 0.00 |
| 21 | 0.04 | 0.00 | 0.06 | 0.00 |
| 28 | 0.05 | 0.00 | 0.06 | 0.00 |
| 42 | 0.06 | 0.00 | 0.07 | 0.01 |
| 56 | 0.06 | 0.00 | 0.07 | 0.01 |

Note:

% Expansion value on $n^{th}$ day =

$$\frac{[(\text{mortar bar reading of } n^{th} \text{ day} - \text{ref. bar reading of } n^{th} \text{ day}) - (\text{mortar bar reading of } 0^{th} \text{ day} - \text{ref. bar reading of } 0^{th} \text{ day})] \times 100}{\text{Original length of the mortar bar}}$$

Table A.4 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with Spratt Limestone and Selected Fly Ashes at 15% Dosage

| | Spratt Limestone- 15% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Potassium Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.162 | 0.006 | 0.090 | 0.009 | 0.052 | 0.004 | 0.271 | 0.009 | 0.074 | 0.006 | 0.019 | 0.005 |
| 7 | 0.237 | 0.002 | 0.141 | 0.009 | 0.080 | 0.004 | 0.405 | 0.007 | 0.151 | 0.009 | 0.032 | 0.002 |
| 11 | 0.281 | 0.005 | 0.164 | 0.007 | 0.099 | 0.004 | 0.503 | 0.014 | 0.196 | 0.007 | 0.043 | 0.001 |
| 14 | 0.338 | 0.005 | 0.187 | 0.011 | 0.115 | 0.002 | 0.593 | 0.020 | 0.236 | 0.013 | 0.051 | 0.002 |
| 21 | 0.518 | 0.008 | 0.293 | 0.016 | 0.199 | 0.004 | 0.772 | 0.009 | 0.374 | 0.021 | 0.089 | 0.006 |
| 28 | 0.799 | 0.012 | 0.543 | 0.027 | 0.383 | 0.008 | 1.005 | 0.071 | 0.593 | 0.032 | 0.150 | 0.011 |
| 42 | 1.065 | 0.041 | 0.715 | 0.034 | 0.530 | 0.010 | 1.225 | 0.029 | 0.742 | 0.042 | 0.195 | 0.013 |
| 56 | 1.290 | 0.023 | 1.011 | 0.033 | 0.801 | 0.011 | 1.405 | 0.033 | 1.091 | 0.077 | 0.251 | 0.024 |

*Note: All Expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table A.5 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with Spratt Limestone and Selected Fly Ashes at 25% Dosage

| | Spratt Limestone- 25% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Potassium Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.125 | 0.007 | 0.043 | 0.003 | 0.025 | 0.001 | 0.152 | 0.009 | -0.001 | 0.000 | -0.003 | 0.001 |
| 7 | 0.212 | 0.006 | 0.064 | 0.004 | 0.039 | 0.001 | 0.380 | 0.012 | 0.006 | 0.001 | -0.004 | 0.001 |
| 11 | 0.283 | 0.007 | 0.090 | 0.003 | 0.053 | 0.002 | 0.567 | 0.019 | 0.020 | 0.000 | -0.003 | 0.002 |
| 14 | 0.344 | 0.007 | 0.090 | 0.003 | 0.080 | 0.005 | 0.678 | 0.024 | 0.039 | 0.002 | 0.000 | 0.001 |
| 21 | 0.569 | 0.012 | 0.222 | 0.003 | 0.158 | 0.008 | 0.951 | 0.050 | 0.067 | 0.003 | 0.020 | 0.003 |
| 28 | 0.874 | 0.012 | 0.368 | 0.004 | 0.265 | 0.012 | 1.262 | 0.077 | 0.108 | 0.006 | 0.047 | 0.003 |
| 42 | 1.126 | 0.050 | 0.526 | 0.003 | 0.393 | 0.014 | 1.484 | 0.091 | 0.158 | 0.012 | 0.073 | 0.003 |
| 56 | 1.296 | 0.035 | 0.743 | 0.004 | 0.577 | 0.016 | 1.660 | 0.096 | 0.240 | 0.025 | 0.109 | 0.006 |

*Note: All Expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table A.6 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with Spratt Limestone and Selected Fly Ashes at 35% Dosage

| | Spratt Limestone- 35% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.036 | 0.001 | 0.008 | 0.001 | 0.002 | 0.001 | -0.005 | 0.002 | -0.012 | 0.003 | -0.005 | 0.002 |
| 7 | 0.075 | 0.007 | 0.017 | 0.002 | 0.004 | 0.004 | 0.079 | 0.004 | -0.016 | 0.003 | -0.013 | 0.002 |
| 11 | 0.112 | 0.002 | 0.029 | 0.001 | 0.014 | 0.001 | 0.122 | 0.011 | -0.020 | 0.003 | -0.015 | 0.002 |
| 14 | 0.155 | 0.001 | 0.045 | 0.001 | 0.022 | 0.001 | 0.209 | 0.021 | -0.020 | 0.003 | -0.018 | 0.002 |
| 21 | 0.236 | 0.003 | 0.087 | 0.005 | 0.037 | 0.004 | 0.338 | 0.021 | -0.016 | 0.004 | -0.017 | 0.002 |
| 28 | 0.458 | 0.011 | 0.185 | 0.007 | 0.151 | 0.037 | 0.540 | 0.027 | -0.010 | 0.002 | -0.008 | 0.004 |
| 42 | 0.741 | 0.018 | 0.321 | 0.008 | 0.246 | 0.003 | 0.742 | 0.029 | 0.009 | 0.005 | 0.008 | 0.006 |
| 56 | 0.895 | 0.020 | 0.403 | 0.009 | 0.320 | 0.002 | 0.825 | 0.031 | 0.020 | 0.006 | 0.013 | 0.006 |

*Note: All Expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table A.7 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with Spratt Limestone and Low and Intermediate Lime Fly Ashes at 25% Dosage

| | Spratt Limestone- 25% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash- LL1 | | Fly Ash-LL2 | | Fly Ash-LL4 | | Fly Ash-LL1 | | Fly Ash-LL2 | | Fly Ash-LL4 | |
| (Day) | Avg. | Std.Dev | Avg. | Std.Dev | Avg. | Std.Dev | Avg. | Std.Dev | Avg. | Std.Dev | Avg. | Std.Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.024 | 0.006 | 0.025 | 0.003 | 0.014 | 0.004 | -0.004 | 0.002 | 0.002 | 0.004 | 0.003 | 0.007 |
| 7 | 0.034 | 0.006 | 0.035 | 0.005 | 0.013 | 0.004 | -0.010 | 0.002 | -0.005 | 0.003 | -0.016 | 0.002 |
| 11 | 0.046 | 0.006 | 0.051 | 0.008 | 0.020 | 0.005 | -0.009 | 0.004 | -0.006 | 0.003 | -0.012 | 0.002 |
| 14 | 0.069 | 0.008 | 0.072 | 0.006 | 0.035 | 0.003 | 0.001 | 0.008 | -0.008 | 0.002 | -0.013 | 0.004 |
| 21 | 0.141 | 0.009 | 0.140 | 0.009 | 0.101 | 0.005 | 0.012 | 0.003 | 0.010 | 0.004 | 0.001 | 0.005 |
| 28 | 0.228 | 0.010 | 0.235 | 0.006 | 0.182 | 0.011 | 0.037 | 0.006 | 0.045 | 0.004 | 0.021 | 0.005 |
| Age | Fly Ash-LL5 | | Fly Ash-IL1 | | Fly Ash-IL2 | | Fly Ash-LL5 | | Fly Ash-IL1 | | Fly Ash-IL2 | |
| (Day) | Avg. | Std.Dev | Avg. | Std.Dev | Avg. | Std.Dev | Avg. | Std.Dev | Avg. | Std.Dev | Avg. | Std.Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.005 | 0.004 | 0.024 | 0.004 | 0.010 | 0.003 | -0.012 | 0.001 | -0.004 | 0.001 | -0.010 | 0.005 |
| 7 | 0.013 | 0.003 | 0.040 | 0.005 | 0.029 | 0.004 | -0.009 | 0.003 | -0.007 | 0.002 | -0.007 | 0.003 |
| 11 | 0.018 | 0.004 | 0.068 | 0.004 | 0.048 | 0.002 | -0.010 | 0.002 | -0.008 | 0.002 | -0.002 | 0.007 |
| 14 | 0.027 | 0.005 | 0.090 | 0.005 | 0.074 | 0.005 | -0.009 | 0.003 | 0.000 | 0.002 | -0.003 | 0.006 |
| 21 | 0.040 | 0.004 | 0.160 | 0.004 | 0.135 | 0.005 | -0.009 | 0.004 | 0.020 | 0.003 | 0.005 | 0.005 |
| 28 | 0.076 | 0.004 | 0.266 | 0.004 | 0.210 | 0.003 | 0.011 | 0.005 | 0.063 | 0.006 | 0.032 | 0.007 |
| Age | Fly Ash-IL3 | | Fly Ash-IL4 | | Fly Ash-IL6 | | Fly Ash-IL3 | | Fly Ash-IL4 | | Fly Ash-IL6 | |
| (Day) | Avg. | Std.Dev | Avg. | Std.Dev | Avg. | Std.Dev | Avg. | Std.Dev | Avg. | Std.Dev | Avg. | Std.Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.015 | 0.002 | 0.012 | 0.001 | 0.034 | 0.004 | -0.012 | 0.001 | -0.009 | 0.004 | -0.011 | 0.002 |
| 7 | 0.024 | 0.001 | 0.032 | 0.005 | 0.065 | 0.006 | -0.013 | 0.003 | -0.006 | 0.001 | -0.005 | 0.006 |
| 11 | 0.038 | 0.004 | 0.056 | 0.001 | 0.102 | 0.006 | -0.014 | 0.001 | -0.004 | 0.003 | 0.006 | 0.005 |
| 14 | 0.077 | 0.002 | 0.087 | 0.005 | 0.131 | 0.007 | -0.009 | 0.001 | -0.004 | 0.003 | 0.012 | 0.007 |
| 21 | 0.148 | 0.001 | 0.155 | 0.004 | 0.212 | 0.009 | 0.002 | 0.008 | 0.013 | 0.002 | 0.053 | 0.008 |
| 28 | 0.206 | 0.002 | 0.233 | 0.004 | 0.300 | 0.014 | 0.017 | 0.003 | 0.028 | 0.003 | 0.117 | 0.029 |

Table A.8 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with Spratt Limestone and High Lime Fly Ashes at 25% Dosage

| | Spratt Limestone- 25% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL1 | | Fly Ash-HL2 | | Fly Ash-HL4 | | Fly Ash-HL1 | | Fly Ash-HL2 | | Fly Ash-HL4 | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.038 | 0.001 | 0.083 | 0.002 | 0.110 | 0.003 | -0.003 | 0.001 | 0.081 | 0.005 | 0.122 | 0.008 |
| 7 | 0.091 | 0.003 | 0.174 | 0.005 | 0.218 | 0.003 | 0.014 | 0.001 | 0.272 | 0.005 | 0.336 | 0.015 |
| 11 | 0.139 | 0.004 | 0.217 | 0.005 | 0.265 | 0.017 | 0.050 | 0.003 | 0.414 | 0.007 | 0.483 | 0.010 |
| 14 | 0.177 | 0.005 | 0.305 | 0.010 | 0.346 | 0.004 | 0.075 | 0.005 | 0.622 | 0.010 | 0.687 | 0.011 |
| 21 | 0.270 | 0.008 | 0.449 | 0.015 | 0.481 | 0.005 | 0.131 | 0.008 | 0.854 | 0.019 | 0.902 | 0.020 |
| 28 | 0.388 | 0.012 | 0.595 | 0.020 | 0.619 | 0.008 | 0.204 | 0.010 | 1.069 | 0.017 | 1.089 | 0.032 |

Table A9 Percentage Expansions of Mortar Bars in Standard and Modified ASTM C 1567 Test for New Mexico Rhyolite with Selected Fly Ashes at 15% Dosage

| | New Mexico- 15% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.467 | 0.074 | 0.128 | 0.002 | 0.038 | 0.003 | 1.227 | 0.070 | 0.350 | 0.015 | 0.407 | 0.025 |
| 7 | 0.848 | 0.079 | 0.347 | 0.006 | 0.216 | 0.012 | 1.761 | 0.036 | 1.393 | 0.027 | 0.921 | 0.051 |
| 11 | 0.889 | 0.083 | 0.386 | 0.006 | 0.255 | 0.013 | 1.773 | 0.036 | 1.420 | 0.028 | 1.319 | 0.049 |
| 14 | 1.048 | 0.089 | 0.507 | 0.010 | 0.374 | 0.012 | 1.792 | 0.036 | 1.446 | 0.029 | 1.381 | 0.048 |
| 21 | 1.148 | 0.069 | 0.643 | 0.015 | 0.503 | 0.015 | 1.857 | 0.037 | 1.455 | 0.032 | 1.417 | 0.071 |
| 28 | 1.246 | 0.061 | 0.773 | 0.018 | 0.634 | 0.018 | 1.865 | 0.036 | 1.458 | 0.032 | 1.441 | 0.072 |
| 42 | 1.364 | 0.058 | 0.979 | 0.021 | 0.856 | 0.019 | 1.884 | 0.036 | 1.476 | 0.032 | 1.448 | 0.073 |
| 56 | 1.42 | 0.06 | 1.10 | 0.02 | 0.99 | 0.02 | 1.89 | 0.04 | 1.48 | 0.03 | 1.46 | 0.07 |

*Note: All Expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table A1.0 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with New Mexico Rhyolite and Selected Fly Ashes at 25% Dosage

| | New Mexico- 25% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.409 | 0.007 | 0.033 | 0.004 | 0.035 | 0.001 | 1.878 | 0.015 | 0.006 | 0.002 | 0.014 | 0.001 |
| 7 | 0.650 | 0.019 | 0.156 | 0.006 | 0.051 | 0.003 | 2.093 | 0.017 | 0.020 | 0.003 | 0.025 | 0.016 |
| 11 | 0.792 | 0.025 | 0.246 | 0.012 | 0.066 | 0.011 | 2.131 | 0.014 | 0.701 | 0.025 | 0.016 | 0.006 |
| 14 | 0.892 | 0.023 | 0.326 | 0.015 | 0.107 | 0.010 | 2.134 | 0.013 | 1.566 | 0.088 | 0.023 | 0.035 |
| 21 | 1.043 | 0.018 | 0.458 | 0.017 | 0.263 | 0.017 | 2.155 | 0.014 | 1.832 | 0.310 | 0.415 | 0.133 |
| 28 | 1.154 | 0.018 | 0.591 | 0.024 | 0.372 | 0.016 | Mortar Bars Broke | | | 1.920 | 0.235 | 0.784 |
| 42 | 1.272 | 0.022 | 0.820 | 0.030 | 0.569 | 0.018 | | | | 1.941 | 0.231 | 0.902 |
| 56 | 1.323 | 0.023 | 0.946 | 0.035 | 0.686 | 0.018 | | | | 1.949 | 0.232 | 0.918 |

*Note: All Expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*


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Table A.11 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with New Mexico Rhyolite and Selected Fly Ashes at 35% Dosage

| | New Mexico- 35% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.104 | 0.008 | 0.017 | 0.001 | 0.004 | 0.001 | 0.835 | 0.071 | -0.001 | 0.001 | 0.004 | 0.002 |
| 7 | 0.242 | 0.009 | 0.043 | 0.002 | 0.020 | 0.002 | 1.881 | 0.059 | 0.006 | 0.006 | 0.004 | 0.002 |
| 11 | 0.348 | 0.015 | 0.092 | 0.008 | 0.033 | 0.001 | 1.975 | 0.058 | 0.390 | 0.038 | 0.000 | 0.002 |
| 14 | 0.385 | 0.013 | 0.108 | 0.007 | 0.038 | 0.002 | 2.039 | 0.057 | 0.942 | 0.083 | 0.000 | 0.001 |
| 21 | 0.438 | 0.016 | 0.219 | 0.010 | 0.085 | 0.002 | 2.089 | 0.027 | 1.318 | 0.089 | 0.015 | 0.002 |
| 28 | 0.618 | 0.028 | 0.270 | 0.010 | 0.126 | 0.005 | 2.055 | 0.028 | | | 0.018 | 0.002 |
| 42 | 0.821 | 0.039 | 0.420 | 0.008 | 0.233 | 0.006 | 2.080 | 0.027 | | | 0.029 | 0.002 |
| 56 | 0.921 | 0.046 | 0.516 | 0.006 | 0.306 | 0.007 | 2.084 | 0.029 | | | 0.037 | 0.002 |

*Note: All Expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table A.12 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with North Carolina Argillite and Selected Fly Ashes at 15% Dosage

| | North Carolina- 15% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.175 | 0.005 | 0.063 | 0.008 | 0.020 | 0.005 | 0.030 | 0.004 | 0.030 | 0.010 | 0.007 | 0.002 |
| 7 | 0.268 | 0.002 | 0.106 | 0.006 | 0.039 | 0.003 | 0.655 | 0.010 | 0.050 | 0.011 | 0.016 | 0.002 |
| 11 | 0.352 | 0.017 | 0.172 | 0.004 | 0.059 | 0.006 | 0.698 | 0.066 | 0.124 | 0.002 | 0.023 | 0.002 |
| 14 | 0.414 | 0.001 | 0.202 | 0.009 | 0.080 | 0.006 | 0.726 | 0.051 | 0.196 | 0.029 | 0.028 | 0.003 |
| 21 | 0.463 | 0.003 | 0.244 | 0.011 | 0.097 | 0.005 | 0.776 | 0.033 | 0.234 | 0.038 | 0.030 | 0.003 |
| 28 | 0.522 | 0.004 | 0.301 | 0.014 | 0.144 | 0.008 | 0.818 | 0.018 | 0.251 | 0.041 | 0.037 | 0.003 |
| 42 | 0.591 | 0.006 | 0.383 | 0.018 | 0.202 | 0.006 | 0.836 | 0.021 | 0.275 | 0.041 | 0.047 | 0.006 |
| 56 | 0.607 | 0.005 | 0.444 | 0.023 | | | | | 0.288 | 0.043 | 0.054 | 0.007 |

*Note: All Expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table A.13 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with North Carolina Argillite and Selected Fly Ashes at 25% Dosage

| | North Carolina- 25% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.157 | 0.003 | 0.014 | 0.002 | 0.021 | 0.004 | 0.089 | 0.004 | -0.004 | 0.002 | 0.004 | 0.001 |
| 7 | 0.310 | 0.006 | 0.033 | 0.002 | 0.032 | 0.002 | 0.637 | 0.036 | -0.002 | 0.002 | 0.003 | 0.001 |
| 11 | 0.351 | 0.007 | 0.045 | 0.001 | 0.040 | 0.002 | 0.687 | 0.044 | 0.043 | 0.049 | 0.005 | 0.001 |
| 14 | 0.424 | 0.007 | 0.071 | 0.001 | 0.050 | 0.003 | 0.716 | 0.043 | 0.013 | 0.003 | 0.013 | 0.001 |
| 21 | 0.456 | 0.011 | 0.091 | 0.003 | 0.060 | 0.002 | 0.716 | 0.052 | 0.019 | 0.002 | 0.021 | 0.001 |
| 28 | 0.505 | 0.014 | 0.124 | 0.008 | 0.072 | 0.002 | 0.731 | 0.050 | 0.023 | 0.004 | 0.026 | 0.003 |
| 42 | 0.569 | 0.013 | 0.183 | 0.011 | 0.095 | 0.003 | 0.756 | 0.050 | 0.034 | 0.002 | 0.040 | 0.002 |
| 56 | 0.607 | 0.014 | 0.241 | 0.011 | 0.124 | 0.005 | 0.776 | 0.051 | 0.044 | 0.003 | 0.050 | 0.002 |

*Note: All Expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table A.14 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with North Carolina Argillite and Selected Fly Ashes at 35% Dosage

| | North Carolina- 35% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.041 | 0.002 | 0.008 | 0.001 | 0.002 | 0.002 | 0.004 | 0.003 | -0.002 | 0.001 | -0.003 | 0.001 |
| 7 | 0.075 | 0.008 | 0.010 | 0.000 | 0.001 | 0.003 | 0.029 | 0.005 | -0.010 | 0.002 | -0.012 | 0.003 |
| 11 | 0.096 | 0.009 | 0.015 | 0.001 | 0.004 | 0.003 | 0.054 | 0.005 | -0.018 | 0.001 | -0.016 | 0.002 |
| 14 | 0.139 | 0.008 | 0.022 | 0.003 | 0.011 | 0.002 | 0.076 | 0.007 | -0.027 | 0.001 | -0.027 | 0.001 |
| 21 | 0.192 | 0.010 | 0.045 | 0.002 | 0.029 | 0.003 | 0.100 | 0.007 | -0.020 | 0.001 | -0.025 | 0.002 |
| 28 | 0.220 | 0.014 | 0.052 | 0.002 | 0.036 | 0.003 | 0.109 | 0.007 | -0.011 | 0.001 | -0.025 | 0.001 |
| 42 | 0.264 | 0.013 | 0.075 | 0.003 | 0.055 | 0.005 | 0.126 | 0.008 | -0.002 | 0.003 | -0.017 | 0.001 |
| 56 | 0.236 | 0.065 | 0.086 | 0.004 | 0.055 | 0.008 | 0.132 | 0.007 | -0.002 | 0.002 | -0.019 | 0.002 |

*Note: All expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table A.15 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with South Dakota Quartzite and Selected Fly Ashes at 15% Dosage

| | South Dakota- 15% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.085 | 0.004 | 0.042 | 0.002 | 0.027 | 0.004 | 0.124 | 0.005 | 0.034 | 0.004 | 0.011 | 0.002 |
| 7 | 0.143 | 0.004 | 0.077 | 0.002 | 0.058 | 0.003 | 0.365 | 0.011 | 0.064 | 0.000 | 0.018 | 0.002 |
| 11 | 0.203 | 0.005 | 0.121 | 0.004 | 0.094 | 0.009 | 0.531 | 0.016 | 0.147 | 0.004 | 0.025 | 0.004 |
| 14 | 0.243 | 0.006 | 0.159 | 0.003 | 0.131 | 0.005 | 0.571 | 0.018 | 0.192 | 0.003 | 0.036 | 0.005 |
| 21 | 0.304 | 0.011 | 0.237 | 0.005 | 0.204 | 0.007 | 0.591 | 0.021 | 0.295 | 0.004 | 0.065 | 0.006 |
| 28 | 0.335 | 0.009 | 0.292 | 0.007 | 0.256 | 0.009 | 0.596 | 0.021 | 0.325 | 0.004 | 0.072 | 0.012 |
| 42 | 0.407 | 0.015 | 0.380 | 0.006 | 0.343 | 0.011 | 0.603 | 0.019 | 0.338 | 0.002 | 0.081 | 0.008 |
| 56 | 0.493 | 0.018 | 0.457 | 0.004 | 0.436 | 0.012 | 0.626 | 0.022 | 0.349 | 0.002 | 0.089 | 0.013 |

*Note: All expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table A.16 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with South Dakota Quartzite and Selected Fly Ashes at 25% Dosage

| | South Dakota- 25% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.080 | 0.003 | 0.013 | 0.005 | 0.027 | 0.005 | 0.116 | 0.004 | 0.007 | 0.001 | 0.016 | 0.001 |
| 7 | 0.141 | 0.003 | 0.049 | 0.002 | 0.041 | 0.004 | 0.313 | 0.003 | 0.012 | 0.000 | 0.014 | 0.001 |
| 11 | 0.181 | 0.003 | 0.068 | 0.002 | 0.052 | 0.005 | 0.462 | 0.005 | 0.025 | 0.001 | 0.016 | 0.001 |
| 14 | 0.217 | 0.004 | 0.095 | 0.001 | 0.062 | 0.005 | 0.499 | 0.006 | 0.038 | 0.004 | 0.016 | 0.001 |
| 21 | 0.275 | 0.007 | 0.131 | 0.004 | 0.083 | 0.004 | 0.520 | 0.007 | 0.060 | 0.007 | 0.024 | 0.003 |
| 28 | 0.327 | 0.006 | 0.169 | 0.004 | 0.105 | 0.002 | 0.524 | 0.006 | 0.068 | 0.009 | 0.033 | 0.004 |
| 42 | 0.397 | 0.006 | 0.231 | 0.004 | 0.146 | 0.002 | 0.539 | 0.014 | 0.079 | 0.009 | 0.046 | 0.005 |
| 56 | 0.450 | 0.006 | 0.284 | 0.007 | 0.178 | 0.004 | 0.558 | 0.006 | 0.086 | 0.009 | 0.051 | 0.005 |

*Note: All expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table A.17 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with South Dakota Quartzite and Selected Fly Ashes at 35% Dosage

| | South Dakota- 35% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.068 | 0.003 | 0.036 | 0.001 | 0.014 | 0.001 | 0.058 | 0.002 | 0.009 | 0.002 | 0.000 | 0.001 |
| 7 | 0.126 | 0.004 | 0.046 | 0.002 | 0.017 | 0.002 | 0.139 | 0.001 | 0.007 | 0.003 | 0.000 | 0.002 |
| 11 | 0.180 | 0.008 | 0.052 | 0.004 | 0.022 | 0.002 | 0.213 | 0.011 | 0.001 | 0.002 | -0.003 | 0.001 |
| 14 | 0.212 | 0.009 | 0.065 | 0.003 | 0.030 | 0.003 | 0.250 | 0.021 | -0.002 | 0.002 | -0.007 | 0.001 |
| 21 | 0.264 | 0.012 | 0.081 | 0.001 | 0.043 | 0.000 | 0.282 | 0.026 | 0.000 | 0.000 | -0.009 | 0.001 |
| 28 | 0.285 | 0.014 | 0.103 | 0.001 | 0.047 | 0.002 | 0.289 | 0.026 | -0.001 | 0.002 | -0.013 | 0.001 |
| 42 | 0.346 | 0.027 | 0.126 | 0.001 | 0.049 | 0.002 | 0.308 | 0.025 | 0.003 | 0.001 | -0.021 | 0.002 |
| 56 | 0.420 | 0.019 | 0.147 | 0.002 | 0.085 | 0.002 | 0.332 | 0.025 | 0.009 | 0.001 | | |

*Note: All expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table A.18 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with Illinois Dolomite and Selected Fly Ashes at 25% Dosage

| | Illinois Dolomite- 25% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.034 | 0.001 | 0.006 | 0.002 | 0.017 | 0.001 | 0.042 | 0.001 | -0.002 | 0.001 | 0.001 | 0.002 |
| 7 | 0.050 | 0.003 | 0.015 | 0.002 | 0.027 | 0.002 | 0.065 | 0.002 | -0.002 | 0.002 | 0.004 | 0.006 |
| 11 | 0.059 | 0.001 | 0.023 | 0.001 | 0.033 | 0.000 | 0.084 | 0.003 | 0.002 | 0.002 | 0.005 | 0.001 |
| 14 | 0.066 | 0.002 | 0.030 | 0.003 | 0.038 | 0.001 | 0.095 | 0.003 | 0.005 | 0.001 | 0.016 | 0.009 |
| 21 | 0.071 | 0.003 | 0.034 | 0.001 | 0.042 | 0.001 | 0.108 | 0.003 | 0.008 | 0.001 | 0.017 | 0.002 |
| 28 | 0.079 | 0.003 | 0.046 | 0.002 | 0.053 | 0.001 | 0.132 | 0.003 | 0.020 | 0.001 | 0.032 | 0.002 |
| 42 | 0.084 | 0.004 | 0.048 | 0.001 | 0.060 | 0.002 | 0.144 | 0.004 | 0.026 | 0.002 | 0.038 | 0.003 |
| 56 | 0.089 | 0.004 | 0.055 | 0.001 | 0.065 | 0.002 | 0.149 | 0.005 | 0.036 | 0.002 | 0.043 | 0.003 |

*Note: All expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table A.19 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with Spratt Limestone and Slag at 40% and 50% Dosage

| | Spratt Limestone | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | 50% Pot. Acetate | | | |
| Age | 40% Slag | | 50% Slag | | 40% Slag | | 50% Slag | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.038 | 0.003 | 0.023 | 0.000 | 0.031 | 0.001 | 0.003 | 0.002 |
| 7 | 0.057 | 0.009 | 0.034 | 0.001 | 0.041 | 0.000 | 0.003 | 0.003 |
| 11 | 0.092 | 0.002 | 0.063 | 0.004 | 0.086 | 0.001 | 0.022 | 0.006 |
| 14 | 0.126 | 0.011 | 0.094 | 0.002 | 0.125 | 0.001 | 0.037 | 0.003 |
| 21 | 0.198 | 0.008 | 0.157 | 0.001 | 0.195 | 0.004 | 0.057 | 0.004 |
| 28 | 0.293 | 0.012 | 0.253 | 0.006 | 0.242 | 0.002 | 0.091 | 0.004 |
| 42 | 0.534 | 0.024 | 0.432 | 0.005 | 0.353 | 0.006 | 0.131 | 0.006 |
| 56 | 0.735 | 0.031 | 0.604 | 0.006 | 0.468 | 0.001 | 0.158 | 0.005 |

*Note: All expansions are in percentage.*

Table A.20 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with New Mexico Rhyolite and Slag at 40% and 50% Dosage

| | New Mexico Rhyolite | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | 50% Pot. Acetate | | | |
| Age | 40% Slag | | 50% Slag | | 40% Slag | | 50% Slag | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.075 | 0.005 | 0.024 | 0.001 | 0.445 | 0.047 | -0.010 | 0.003 |
| 7 | 0.221 | 0.006 | 0.041 | 0.003 | 1.040 | 0.086 | -0.009 | 0.004 |
| 11 | 0.328 | 0.006 | 0.102 | 0.005 | 1.194 | 0.080 | -0.005 | 0.003 |
| 14 | 0.385 | 0.006 | 0.147 | 0.005 | 1.225 | 0.078 | 0.007 | 0.004 |
| 21 | 0.556 | 0.011 | 0.220 | 0.004 | 1.245 | 0.077 | 0.014 | 0.006 |
| 28 | 0.686 | 0.010 | 0.319 | 0.006 | 1.255 | 0.078 | 0.025 | 0.006 |
| 42 | 0.913 | 0.018 | 0.447 | 0.003 | 1.258 | 0.107 | 0.037 | 0.006 |
| 56 | 1.076 | 0.021 | 0.574 | 0.005 | 1.270 | 0.110 | 0.046 | 0.004 |

*Note: All expansions are in percentage.*

Table A.21 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with North Carolina Argillite and Slag at 40% and 50% Dosage

| | North Carolina Argillite | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | 50% Pot. Acetate | | | |
| Age | 40% Slag | | 50% Slag | | 40% Slag | | 50% Slag | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.028 | 0.006 | 0.022 | 0.002 | 0.018 | 0.006 | -0.006 | 0.005 |
| 7 | 0.051 | 0.009 | 0.028 | 0.002 | 0.047 | 0.011 | -0.010 | 0.004 |
| 11 | 0.083 | 0.008 | 0.020 | 0.003 | 0.071 | 0.016 | -0.008 | 0.005 |
| 14 | 0.113 | 0.011 | 0.014 | 0.007 | 0.077 | 0.019 | -0.001 | 0.002 |
| 21 | 0.166 | 0.011 | 0.044 | 0.005 | 0.089 | 0.017 | 0.003 | 0.001 |
| 28 | 0.199 | 0.009 | 0.067 | 0.004 | 0.093 | 0.008 | 0.012 | 0.001 |
| 42 | 0.265 | 0.004 | 0.102 | 0.007 | 0.109 | 0.012 | 0.022 | 0.002 |
| 56 | 0.333 | 0.013 | 0.144 | 0.012 | 0.119 | 0.016 | 0.027 | 0.002 |

*Note: All expansions are in percentage.*

Table A.22 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with South Dakota Quartzite and Slag at 40% and 50% Dosage

| | South Dakota Quartzite | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | 50% Pot. Acetate | | | |
| Age | 40% Slag | | 50% Slag | | 40% Slag | | 50% Slag | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.025 | 0.003 | 0.025 | 0.002 | 0.038 | 0.002 | 0.004 | 0.002 |
| 7 | 0.081 | 0.005 | 0.042 | 0.003 | 0.161 | 0.010 | 0.024 | 0.003 |
| 11 | 0.114 | 0.006 | 0.060 | 0.007 | 0.317 | 0.018 | 0.036 | 0.004 |
| 14 | 0.127 | 0.008 | 0.077 | 0.005 | 0.407 | 0.015 | 0.051 | 0.004 |
| 21 | 0.203 | 0.009 | 0.092 | 0.004 | 0.526 | 0.010 | 0.059 | 0.007 |
| 28 | 0.255 | 0.014 | 0.132 | 0.004 | 0.536 | 0.009 | 0.070 | 0.006 |
| 42 | 0.331 | 0.017 | 0.184 | 0.007 | 0.549 | 0.010 | 0.080 | 0.008 |
| 56 | 0.422 | 0.019 | 0.241 | 0.006 | 0.555 | 0.010 | 0.086 | 0.007 |

*Note: All expansions are in percentage.*

Table A.23 Expansions (%) of Mortar Bars in Standard and Modified ASTM C 1567 Test with Illinois Dolomite and Slag at 40% Dosage

| | Illinois Dolomite | | | |
|---|---|---|---|---|
| | 1N NaOH | | 50% Pot. Acetate | |
| Age | 40% Slag | | 50% Slag | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 |
| 3 | 0.002 | 0.001 | 0.011 | 0.001 |
| 7 | 0.017 | 0.001 | 0.024 | 0.001 |
| 11 | 0.007 | 0.002 | 0.008 | 0.002 |
| 14 | 0.027 | 0.002 | 0.028 | 0.001 |
| 21 | 0.044 | 0.002 | 0.038 | 0.002 |
| 28 | 0.047 | 0.002 | 0.035 | 0.001 |
| 42 | 0.066 | 0.040 | 0.047 | 0.001 |
| 56 | 0.081 | 0.042 | 0.052 | 0.000 |

*Note: All expansions are in percentage.*

<u>APPENDIX-B LENGTH CHANGE DATA FOR CONCRETE PRISMS</u>

Table B.1 Expansions (%) of Concrete Prisms in Modified ASTM C 1293 Tests with Spratt Limestone and Selected Fly Ashes at 25% Dosage

| | Spratt Limestone- 25% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Days) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.004 | 0.000 | 0.002 | 0.002 | 0.008 | 0.003 | 0.002 | 0.005 | 0.003 | -0.004 | 0.004 |
| 7 | 0.000 | 0.005 | -0.002 | 0.004 | 0.000 | 0.004 | 0.003 | 0.002 | 0.000 | 0.004 | -0.004 | 0.003 |
| 28 | 0.013 | 0.002 | 0.003 | 0.004 | 0.004 | 0.003 | 0.026 | 0.013 | 0.006 | 0.004 | 0.002 | 0.001 |
| 56 | 0.030 | 0.002 | 0.005 | 0.005 | 0.003 | 0.003 | 0.132 | 0.029 | 0.002 | 0.003 | 0.002 | 0.002 |
| 90 | 0.036 | 0.004 | 0.003 | 0.004 | 0.003 | 0.004 | 0.218 | 0.060 | 0.000 | 0.001 | 0.001 | 0.001 |
| 120 | 0.044 | 0.004 | 0.008 | 0.005 | 0.001 | 0.004 | 0.367 | 0.092 | 0.000 | 0.004 | 0.000 | 0.001 |
| 150 | 0.057 | 0.002 | 0.016 | 0.005 | 0.004 | 0.004 | 0.607 | 0.140 | 0.004 | 0.003 | 0.002 | 0.002 |
| 180 | 0.068 | 0.002 | 0.018 | 0.003 | -0.002 | 0.003 | 0.825 | 0.195 | 0.001 | 0.003 | 0.001 | 0.001 |
| 210 | 0.081 | 0.006 | 0.026 | 0.004 | 0.003 | 0.002 | 1.158 | 0.299 | 0.007 | 0.001 | 0.001 | 0.003 |
| 240 | 0.097 | 0.006 | 0.036 | 0.004 | 0.004 | 0.001 | 1.621 | 0.399 | 0.018 | 0.001 | 0.001 | 0.002 |
| 270 | 0.105 | 0.008 | 0.041 | 0.004 | 0.006 | 0.001 | 2.073 | 0.403 | 0.027 | 0.005 | 0.000 | 0.001 |
| 300 | 0.113 | 0.008 | 0.049 | 0.005 | 0.004 | 0.002 | 2.128 | 0.583 | 0.046 | 0.010 | -0.001 | 0.002 |
| 330 | 0.119 | 0.008 | 0.051 | 0.004 | 0.008 | 0.001 | 2.306 | 0.443 | 0.095 | 0.021 | 0.002 | 0.003 |
| 365 | 0.000 | 0.004 | 0.000 | 0.002 | 0.002 | 0.008 | 0.003 | 0.002 | 0.005 | 0.003 | -0.004 | 0.004 |

*Note: All Expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table B.2 Expansions (%) of Concrete Prisms in Modified ASTM C 1293 Tests with Spratt Limestone and Selected Fly Ashes at 35% Dosage

| | Spratt Limestone- 35% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Days) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0 | 0 | 0 | 0 | 0.00 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 7 | -0.002 | 0.002 | -0.001 | 0.001 | 0.011 | 0.001 | 0.000 | 0.001 | -0.002 | 0.003 | 0.001 | 0.011 |
| 28 | -0.004 | 0.001 | 0.000 | 0.001 | 0.003 | 0.002 | 0.000 | 0.002 | -0.002 | 0.003 | 0.003 | 0.003 |
| 56 | 0.006 | 0.009 | 0.013 | 0.002 | 0.004 | 0.005 | 0.013 | 0.004 | 0.007 | 0.001 | 0.007 | 0.004 |
| 90 | 0.014 | 0.004 | 0.002 | 0.002 | 0.004 | 0.005 | 0.106 | 0.042 | -0.004 | 0.004 | 0.003 | 0.001 |
| 120 | 0.017 | 0.006 | 0.001 | 0.001 | -0.001 | 0.005 | 0.145 | 0.042 | -0.004 | 0.004 | 0.001 | 0.004 |
| 150 | 0.024 | 0.004 | 0.002 | 0.003 | 0.001 | 0.005 | 0.300 | 0.073 | -0.003 | 0.002 | 0.002 | 0.002 |
| 180 | 0.036 | 0.006 | 0.010 | 0.002 | 0.003 | 0.007 | 0.555 | 0.166 | -0.002 | 0.001 | 0.000 | 0.003 |
| 210 | 0.048 | 0.006 | 0.009 | 0.003 | 0.001 | 0.001 | 0.843 | 0.285 | -0.004 | 0.002 | -0.001 | 0.005 |
| 240 | 0.053 | 0.004 | 0.014 | 0.003 | 0.003 | 0.004 | 1.286 | 0.384 | -0.003 | 0.002 | 0.004 | 0.005 |
| 270 | 0.072 | 0.006 | 0.019 | 0.002 | 0.001 | 0.008 | 1.602 | 0.119 | 0.000 | 0.001 | 0.004 | 0.004 |
| 300 | 0.079 | 0.006 | 0.027 | 0.001 | 0.003 | 0.007 | Prisms Cracked and broken | | 0.003 | 0.001 | 0.006 | 0.004 |
| 330 | 0.086 | 0.008 | 0.033 | 0.003 | 0.002 | 0.007 | | | 0.006 | 0.001 | 0.005 | 0.004 |
| 365 | 0.092 | 0.009 | 0.035 | 0.004 | 0.003 | 0.008 | | | 0.007 | 0.001 | 0.007 | 0.005 |

*Note: All Expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table B.3 Expansions (%) of Concrete Prisms in Modified ASTM C 1293 Tests with New Mexico Rhyolite and Selected Fly Ashes at 25% Dosage

| | New Mexico Rhyolite- 25% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Days) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 7 | 0.004 | 0.003 | -0.005 | 0.004 | -0.004 | 0.006 | 0.001 | 0.010 | 0.008 | 0.002 | 0.001 | 0.004 |
| 28 | 0.043 | 0.004 | 0.004 | 0.005 | 0.002 | 0.006 | 0.248 | 0.034 | 0.058 | 0.019 | 0.063 | 0.028 |
| 56 | 0.047 | 0.004 | 0.011 | 0.005 | 0.001 | 0.006 | 1.076 | 0.034 | 0.316 | 0.052 | 0.146 | 0.027 |
| 90 | 0.071 | 0.003 | 0.010 | 0.003 | 0.000 | 0.001 | 2.067 | 0.096 | 0.462 | 0.054 | 0.206 | 0.051 |
| 120 | 0.084 | 0.002 | 0.012 | 0.003 | -0.001 | 0.001 | 2.606 | | 0.665 | 0.072 | 0.264 | 0.071 |
| 150 | 0.102 | 0.002 | 0.014 | 0.003 | 0.001 | 0.001 | Prisms Cracked and Broken. Cannot Take readings | | 0.900 | 0.087 | 0.647 | 0.143 |
| 180 | 0.126 | 0.001 | 0.015 | 0.002 | -0.005 | 0.006 | | | 1.045 | 0.105 | 0.826 | 0.177 |
| 210 | 0.133 | 0.001 | 0.021 | 0.002 | -0.011 | 0.006 | | | 1.261 | 0.072 | 0.955 | 0.190 |
| 240 | 0.146 | 0.002 | 0.028 | 0.002 | -0.007 | 0.006 | | | 1.448 | 0.112 | 1.149 | 0.193 |
| 270 | 0.171 | 0.006 | 0.033 | 0.003 | -0.004 | 0.007 | | | 1.573 | 0.134 | 1.402 | 0.183 |
| 300 | 0.185 | 0.004 | 0.041 | 0.003 | 0.000 | 0.006 | | | 1.699 | 0.131 | 1.582 | 0.234 |
| 330 | 0.195 | 0.004 | 0.046 | 0.003 | 0.005 | 0.004 | | | 1.754 | 0.140 | 1.809 | 0.087 |
| 365 | 0.220 | 0.004 | 0.055 | 0.008 | -0.001 | 0.005 | | | 1.826 | 0.134 | 1.937 | 0.110 |

*Note: All Expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table B.4 Expansions (%) of Concrete Prisms in Modified ASTM C 1293 Tests with New Mexico Rhyolite and Selected Fly Ashes at 35% Dosage

| | New Mexico Rhyolite- 35% Fly Ash | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 N NaOH | | | | | | 50% Pot. Acetate | | | | | |
| Age | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | | Fly Ash-HL3 | | Fly Ash-IL5 | | Fly Ash-LL3 | |
| (Days) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 7 | 0.004 | 0.003 | 0.001 | 0.002 | 0.002 | 0.001 | 0.012 | 0.003 | 0.003 | 0.004 | -0.001 | 0.002 |
| 28 | 0.034 | 0.006 | 0.010 | 0.003 | 0.003 | 0.001 | 0.240 | 0.055 | 0.011 | 0.003 | -0.001 | 0.002 |
| 56 | 0.048 | 0.006 | 0.011 | 0.003 | 0.009 | 0.001 | 0.892 | 0.025 | 0.091 | 0.014 | 0.024 | 0.010 |
| 90 | 0.062 | 0.004 | 0.012 | 0.003 | 0.004 | 0.002 | 1.806 | 0.058 | 0.202 | 0.014 | 0.048 | 0.016 |
| 120 | 0.070 | 0.006 | 0.013 | 0.002 | 0.001 | 0.002 | 2.547 | 0.687 | 0.326 | 0.016 | 0.084 | 0.036 |
| 150 | 0.076 | 0.005 | 0.016 | 0.001 | 0.004 | 0.001 | Prisms Cracked and Broken. Cannot Take readings | | 0.500 | 0.022 | 0.266 | 0.077 |
| 180 | 0.086 | 0.005 | 0.019 | 0.002 | 0.001 | 0.001 | | | 0.708 | 0.038 | 0.392 | 0.096 |
| 210 | 0.093 | 0.003 | 0.021 | 0.002 | -0.005 | 0.001 | | | 0.857 | 0.027 | 0.480 | 0.110 |
| 240 | 0.101 | 0.002 | 0.022 | 0.003 | 0.000 | 0.001 | | | 1.065 | 0.039 | 0.643 | 0.114 |
| 270 | 0.116 | 0.002 | 0.025 | 0.006 | 0.002 | 0.001 | | | 1.336 | 0.051 | 0.817 | 0.098 |
| 300 | 0.130 | 0.003 | 0.032 | 0.007 | 0.007 | 0.002 | | | 1.336 | 0.063 | 0.952 | 0.099 |
| 330 | 0.138 | 0.003 | 0.035 | 0.008 | 0.006 | 0.001 | | | 1.401 | 0.070 | 1.079 | 0.117 |
| 365 | 0.160 | 0.004 | 0.039 | 0.008 | 0.004 | 0.002 | | | 1.513 | 0.069 | 1.217 | 0.126 |

*Note: All Expansions are in percentage. HL-High Lime Fly Ash, IL-Intermediate Lime Fly Ash and LL-Low Lime Fly Ash*

Table B.5 Expansions (%) of Concrete Prisms in Modified ASTM C 1293 Tests with Spratt Limestone and Slag at 40% and 50% Dosage

| | Spratt Limestone-Slag | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 1N NaOH | | | | 50% Pot. Acetate | | | |
| Age | 40% Slag | | 50% Slag | | 40% Slag | | 50% Slag | |
| (Days) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 7 | 0.000 | 0.005 | 0.005 | 0.003 | 0.000 | 0.004 | 0.010 | 0.003 |
| 28 | 0.006 | 0.006 | 0.008 | 0.005 | 0.004 | 0.005 | 0.008 | 0.003 |
| 56 | 0.008 | 0.008 | 0.009 | 0.007 | 0.004 | 0.004 | 0.010 | 0.002 |
| 90 | 0.006 | 0.001 | 0.012 | 0.005 | 0.002 | 0.004 | 0.011 | 0.003 |
| 120 | 0.009 | 0.008 | 0.011 | 0.005 | 0.001 | 0.002 | 0.012 | 0.002 |
| 180 | 0.016 | 0.007 | 0.013 | 0.005 | 0.007 | 0.006 | 0.010 | 0.004 |
| 210 | 0.015 | 0.005 | 0.012 | 0.006 | 0.003 | 0.004 | 0.008 | 0.002 |
| 240 | 0.013 | 0.005 | 0.009 | 0.006 | 0.004 | 0.003 | 0.007 | 0.002 |
| 270 | 0.017 | 0.005 | 0.012 | 0.005 | 0.007 | 0.003 | 0.008 | 0.002 |
| 300 | 0.024 | 0.005 | 0.017 | 0.004 | 0.016 | 0.005 | 0.012 | 0.002 |
| 365 | 0.038 | 0.004 | 0.023 | 0.003 | 0.018 | 0.004 | 0.040 | 0.003 |
| 441 | 0.045 | 0.006 | 0.033 | 0.004 | 0.181 | 0.042 | 0.058 | 0.020 |

Table B.6 Expansions (%) of Concrete Prisms in Modified ASTM C 1293 Tests with New Mexico Rhyolite and Slag at 40% and 50% Dosage

| | New Mexico Rhyolite- Slag | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 1 N NaOH | | | | 50% Pot. Acetate | | | |
| Age | 40% Slag | | 50% Slag | | 40% Slag | | 50% Slag | |
| (Day) | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 7 | -0.004 | 0.003 | 0.005 | 0.001 | -0.003 | 0.002 | -0.003 | 0.005 |
| 28 | -0.002 | 0.003 | 0.007 | 0.003 | -0.001 | 0.001 | -0.004 | 0.005 |
| 56 | 0.001 | 0.004 | 0.012 | 0.002 | 0.018 | 0.001 | 0.000 | 0.003 |
| 90 | 0.008 | 0.004 | 0.013 | 0.002 | 0.170 | 0.027 | 0.029 | 0.004 |
| 120 | 0.007 | 0.004 | 0.013 | 0.002 | 0.452 | 0.068 | 0.155 | 0.013 |
| 180 | 0.008 | 0.004 | 0.012 | 0.002 | 0.819 | 0.117 | 0.307 | 0.033 |
| 210 | 0.012 | 0.005 | 0.013 | 0.001 | 1.239 | 0.131 | 0.527 | 0.018 |
| 240 | 0.016 | 0.003 | 0.014 | 0.002 | 1.389 | 0.121 | 0.609 | 0.018 |
| 270 | 0.012 | 0.003 | 0.007 | 0.002 | 1.545 | 0.104 | 0.713 | 0.033 |
| 300 | 0.023 | 0.002 | 0.015 | 0.003 | 1.651 | 0.076 | 0.811 | 0.058 |
| 330 | 0.033 | 0.002 | 0.023 | 0.001 | 1.661 | 0.076 | 0.867 | 0.076 |
| 365 | 0.042 | 0.005 | 0.021 | 0.004 | 1.806 | 0.049 | 1.077 | 0.097 |

*Note: All Expansions are in percentage.*

Table B.7 Expansions (%) of Concrete Prisms in Modified ASTM C 1293 Tests with Illinois Dolomite and Slag at 40% Dosage

| | Illinois Dolomite-Slag | | | |
|---|---|---|---|---|
| | 1 N NaOH | | Pot. Acetate | |
| Age | 40% Slag | | 40% Slag | |
| (Days) | Avg. | Std. Dev | Avg. | Std. Dev |
| 0 | 0.000 | 0.000 | 0.000 | 0.000 |
| 7 | 0.017 | 0.004 | 0.000 | 0.006 |
| 28 | 0.018 | 0.005 | 0.000 | 0.002 |
| 56 | 0.020 | 0.005 | -0.002 | 0.002 |
| 90 | 0.021 | 0.005 | -0.002 | 0.001 |
| 120 | 0.020 | 0.004 | -0.002 | 0.003 |
| 150 | 0.016 | 0.002 | -0.003 | 0.003 |
| 180 | 0.021 | 0.004 | -0.003 | 0.001 |
| 210 | 0.018 | 0.001 | -0.004 | 0.001 |
| 240 | 0.016 | 0.001 | -0.004 | 0.001 |
| 270 | 0.018 | 0.002 | -0.004 | 0.002 |
| 300 | 0.015 | 0.009 | -0.003 | 0.006 |
| 330 | 0.018 | 0.010 | 0.003 | 0.003 |
| 365 | 0.023 | 0.010 | 0.002 | 0.002 |

*Note: All Expansions are in percentage.*

## APPENDIX-C DYNAMIC MODULUS OF ELASTICITY(DME) DATA

Table C.1 DME Data of Concrete Prisms in Modified ASTM C 1293 Tests with Spratt Limestone and High Lime Fly Ash (HL3) at 25% and 35% Dosage

| SPRATT 25% HIGH LIME FLY ASH (HL3) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 6.151 | 6.137 | 6.319 | 6.134 | 6.185 | 0.089 | 6.018 | 6.153 | 5.716 | 5.949 | 5.959 | 0.183 |
| 28 | 7.665 | 7.674 | 7.823 | 7.610 | 7.693 | 0.091 | 6.992 | 5.992 | 6.646 | 6.858 | 6.622 | 0.444 |
| 69 | 7.765 | 7.463 | 7.691 | 7.465 | 7.596 | 0.155 | 6.517 | 6.882 | 6.410 | 6.625 | 6.609 | 0.202 |
| 120 | 7.402 | 7.253 | 7.473 | 7.250 | 7.345 | 0.111 | 5.378 | 5.913 | 5.740 | 5.390 | 5.605 | 0.265 |
| 150 | 7.440 | 7.259 | 7.349 | 7.091 | 7.285 | 0.149 | 4.617 | 5.209 | 5.400 | 4.544 | 4.943 | 0.426 |
| 180 | 7.355 | 7.166 | 7.382 | 7.208 | 7.278 | 0.107 | 2.736 | 3.362 | 4.200 | 2.590 | 3.222 | 0.733 |
| 210 | 7.191 | 7.023 | 7.237 | | 7.150 | 0.113 | 1.840 | 2.344 | 3.356 | | 2.513 | 0.772 |
| 240 | 7.125 | 6.989 | 7.167 | | 7.094 | 0.093 | 1.306 | 0.790 | 2.535 | | 1.544 | 0.896 |
| 270 | 7.073 | 6.934 | 7.097 | | 7.035 | 0.088 | 0.400 | 0.245 | 1.266 | | 0.637 | 0.550 |
| 330 | 7.125 | 6.916 | 7.100 | | 7.047 | 0.114 | 0.110 | Broke | 0.125 | | 0.118 | 0.011 |
| 365 | 7.036 | 6.887 | 7.032 | | 6.985 | 0.085 | 0.088 | Broke | 0.126 | | 0.107 | 0.027 |
| SPRATT 25% HIGH LIME FLY ASH (HL3) | | | | | | | | | | | | |
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 5.945 | 5.760 | 5.764 | 6.082 | 5.888 | 0.156 | 5.933 | 5.848 | 5.909 | 5.720 | 5.852 | 0.095 |
| 28 | 7.725 | 7.568 | 7.488 | 7.782 | 7.641 | 0.136 | 6.927 | 6.800 | 6.901 | 6.677 | 6.826 | 0.114 |
| 69 | 7.683 | 7.536 | 7.486 | 7.784 | 7.622 | 0.137 | 6.713 | 6.619 | 6.697 | 6.493 | 6.631 | 0.100 |
| 120 | 7.734 | 7.397 | 7.336 | 7.195 | 7.415 | 0.228 | 5.675 | 5.710 | 6.075 | 5.586 | 5.761 | 0.215 |
| 150 | 7.479 | 7.365 | 7.277 | 7.559 | 7.420 | 0.124 | 5.762 | 5.001 | 5.403 | 5.027 | 5.298 | 0.360 |
| 180 | 7.599 | 7.513 | 7.353 | 7.729 | 7.549 | 0.158 | 4.056 | 3.722 | 3.921 | 3.665 | 3.841 | 0.180 |
| 210 | 7.367 | 7.234 | 7.153 | | 7.251 | 0.108 | 0.891 | 2.356 | 0.634 | | 1.294 | 0.929 |
| 240 | 7.305 | 7.184 | 7.112 | | 7.201 | 0.098 | 0.138 | 1.528 | 0.415 | | 0.693 | 0.736 |
| 270 | 7.308 | 7.128 | 7.020 | | 7.152 | 0.145 | broke | 0.400 | 0.469 | | 0.434 | 0.049 |
| 330 | 7.230 | 7.092 | 6.969 | | 7.097 | 0.130 | broke | broke | broke | | broke | broke |
| 365 | 7.192 | 7.055 | 6.942 | | 7.063 | 0.125 | broke | broke | broke | | broke | broke |

Note: Prism#4 (1N NaOH and Pot. Acetate) sliced at 180 day test age for SEM studies.

Table C.2 DME Data of Concrete Prisms in Modified ASTM C 1293 Tests with Spratt Limestone and Intermediate Lime Fly Ash (IL5) at 25% and 35% Dosage

| SPRATT 25% INTERMEDIATE LIME FLY ASH (IL5) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 5.748 | 5.798 | 5.507 | 5.796 | 5.712 | 0.139 | 5.546 | 5.876 | 5.567 | 5.945 | 5.734 | 0.206 |
| 7 | 7.416 | 7.415 | 7.207 | 7.345 | 7.346 | 0.098 | 6.721 | 6.829 | 6.672 | 6.869 | 6.773 | 0.092 |
| 28 | 7.681 | 7.705 | 7.467 | 7.612 | 7.616 | 0.107 | 6.837 | 6.981 | 6.948 | 7.015 | 6.945 | 0.077 |
| 71 | 7.681 | 7.731 | 7.620 | 7.648 | 7.670 | 0.048 | 6.941 | 7.090 | 6.931 | 7.128 | 7.023 | 0.101 |
| 120 | 7.690 | 7.715 | 7.501 | 7.612 | 7.629 | 0.096 | 7.051 | 7.238 | 7.114 | 7.257 | 7.165 | 0.099 |
| 150 | 7.630 | 7.689 | 7.452 | 7.557 | 7.582 | 0.102 | 6.999 | 7.206 | 7.091 | 7.266 | 7.140 | 0.119 |
| 180 | 7.727 | 7.693 | 7.530 | 7.659 | 7.652 | 0.086 | 6.989 | 7.493 | 7.141 | 7.463 | 7.271 | 0.246 |
| 210 | 7.530 | 7.545 | 7.351 | | 7.475 | 0.108 | 7.131 | 7.354 | 7.114 | | 7.200 | 0.134 |
| 240 | 7.401 | 7.503 | 7.393 | | 7.433 | 0.061 | 6.952 | 7.126 | 7.014 | | 7.030 | 0.088 |
| 270 | 7.341 | 7.421 | 7.484 | | 7.415 | 0.072 | 6.876 | 7.104 | 6.915 | | 6.965 | 0.122 |
| 300 | 7.368 | 7.406 | 7.255 | | 7.343 | 0.079 | 6.790 | 7.063 | 6.884 | | 6.912 | 0.139 |
| 330 | 7.344 | 7.024 | 7.382 | | 7.250 | 0.197 | 6.671 | 6.927 | 6.783 | | 6.794 | 0.128 |
| 365 | 5.846 | 4.902 | 4.876 | | 5.208 | 0.553 | 5.041 | 6.518 | 5.418 | | 5.659 | 0.768 |
| SPRATT 35% INTERMEDIATE LIME FLY ASH (IL5) | | | | | | | | | | | | |
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 5.651 | 5.548 | 5.322 | 5.603 | 5.531 | 0.145 | 5.355 | 5.787 | 5.453 | 5.322 | 5.479 | 0.213 |
| 7 | 7.546 | 7.214 | 7.231 | 7.459 | 7.363 | 0.166 | 6.637 | 6.861 | 6.648 | 6.449 | 6.649 | 0.169 |
| 28 | 7.785 | 7.705 | 7.596 | 7.680 | 7.691 | 0.078 | 6.764 | 6.960 | 6.748 | 6.542 | 6.754 | 0.171 |
| 71 | 7.592 | 7.513 | 7.493 | 7.725 | 7.581 | 0.105 | 6.653 | 7.051 | 6.869 | 6.663 | 6.809 | 0.190 |
| 120 | 7.585 | 7.530 | 7.461 | 7.720 | 7.574 | 0.110 | 6.923 | 7.272 | 7.030 | 6.844 | 7.017 | 0.186 |
| 150 | 7.554 | 7.736 | 7.522 | 7.686 | 7.624 | 0.103 | 6.890 | 7.321 | 7.098 | 6.897 | 7.052 | 0.204 |
| 180 | 7.772 | 7.461 | 7.592 | 7.861 | 7.671 | 0.180 | 7.118 | 7.374 | 7.264 | 6.807 | 7.141 | 0.246 |
| 210 | 7.487 | 7.624 | 7.400 | | 7.503 | 0.113 | 7.062 | 7.390 | 7.153 | | 7.202 | 0.169 |
| 240 | 7.412 | 7.319 | 7.409 | | 7.380 | 0.053 | 6.895 | 7.267 | 7.084 | | 7.082 | 0.186 |
| 270 | 7.352 | 7.498 | 7.321 | | 7.390 | 0.094 | 6.820 | 7.283 | 7.039 | | 7.048 | 0.232 |
| 300 | 7.341 | 7.222 | 7.320 | | 7.294 | 0.064 | 6.832 | 7.297 | 7.054 | | 7.061 | 0.232 |
| 330 | 7.347 | 7.225 | 7.247 | | 7.273 | 0.065 | 6.768 | 7.283 | 7.153 | | 7.068 | 0.268 |
| 365 | 7.306 | 6.856 | 6.887 | | 7.016 | 0.251 | 6.738 | 6.582 | 6.669 | | 6.663 | 0.078 |

Note: Prism#4 (1N NaOH and Pot. Acetate) sliced at 180 day test age for SEM studies.

Table C.3 DME Data of Concrete Prisms in Modified ASTM C 1293 Tests with Spratt Limestone and Low Lime Fly Ash (LL3) at 25% and 35% Dosage

| SPRATT 25% LOW LIME FLY ASH (LL3) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| (Days) | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 5.700 | 5.577 | 5.749 | 5.635 | 5.665 | 0.075 | 5.706 | 5.491 | 5.722 | 5.619 | 5.634 | 0.106 |
| 7 | 7.443 | 7.284 | 7.542 | 7.355 | 7.406 | 0.112 | 6.758 | 6.562 | 6.722 | 7.150 | 6.798 | 0.250 |
| 28 | 7.666 | 7.506 | 7.769 | 7.586 | 7.632 | 0.112 | 6.901 | 6.788 | 6.891 | 6.874 | 6.863 | 0.052 |
| 70 | 7.720 | 7.629 | 7.825 | 7.603 | 7.694 | 0.100 | 6.932 | 6.930 | 6.660 | 6.651 | 6.793 | 0.159 |
| 120 | 7.750 | 7.600 | 7.784 | 7.674 | 7.702 | 0.082 | 7.111 | 6.966 | 7.111 | 7.129 | 7.079 | 0.076 |
| 150 | 7.750 | 7.614 | 7.833 | 7.673 | 7.718 | 0.095 | 7.169 | 6.998 | 7.159 | 7.186 | 7.128 | 0.087 |
| 180 | 7.876 | 7.683 | 7.794 | 7.932 | 7.821 | 0.108 | 7.193 | 7.007 | 7.242 | 7.342 | 7.196 | 0.140 |
| 210 | 7.764 | 7.592 | 7.859 | | 7.738 | 0.136 | 7.198 | 7.028 | 7.176 | | 7.134 | 0.093 |
| 270 | 7.735 | 7.600 | 7.837 | | 7.724 | 0.119 | 7.139 | 6.946 | 7.094 | | 7.060 | 0.101 |
| 300 | 7.723 | 7.562 | 7.815 | | 7.700 | 0.128 | 7.100 | 7.017 | 7.077 | | 7.065 | 0.043 |
| 330 | 7.708 | 7.677 | 7.738 | | 7.708 | 0.030 | 7.127 | 6.914 | 6.967 | | 7.003 | 0.111 |
| 365 | 7.694 | 7.557 | 7.801 | | 7.684 | 0.123 | 7.094 | 6.865 | 6.892 | | 6.950 | 0.125 |

Note: Prism#4 (1N NaOH and Pot. Acetate) sliced at 180 day test age for SEM studies.

| SPRATT 35% LOW LIME FLY ASH (LL3) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Age | 1N NaOH (DME in psi x 10^6) | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| (Days) | #1 | #2 | #3 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 5.231 | 5.365 | 5.400 | 5.332 | 0.089 | 5.584 | 5.682 | 5.003 | 4.744 | 5.253 | 0.453 |
| 7 | 7.173 | 7.348 | 7.293 | 7.271 | 0.089 | 6.711 | 6.691 | 6.383 | 6.434 | 6.555 | 0.170 |
| 28 | 7.352 | 7.514 | 7.500 | 7.456 | 0.090 | 6.837 | 6.909 | 6.711 | 6.515 | 6.743 | 0.173 |
| 70 | 7.446 | 7.642 | 7.560 | 7.550 | 0.098 | 6.932 | 6.930 | 6.660 | 6.651 | 6.793 | 0.159 |
| 120 | 7.554 | 7.670 | 7.582 | 7.602 | 0.061 | 7.106 | 7.220 | 6.797 | 6.818 | 6.985 | 0.211 |
| 150 | 7.568 | 7.637 | 7.586 | 7.597 | 0.036 | 7.153 | 7.144 | 6.897 | 6.847 | 7.010 | 0.161 |
| 180 | 7.485 | 7.835 | 7.920 | 7.747 | 0.230 | 7.224 | 7.168 | 6.958 | 7.071 | 7.105 | 0.117 |
| 210 | 7.415 | 7.661 | | 7.538 | 0.174 | 7.147 | 7.163 | 6.867 | | 7.059 | 0.166 |
| 270 | 7.444 | 7.643 | | 7.544 | 0.141 | 7.070 | 7.132 | 6.835 | | 7.012 | 0.157 |
| 300 | 7.430 | 7.617 | | 7.524 | 0.132 | 7.040 | 7.033 | 6.866 | | 6.980 | 0.098 |
| 330 | 7.498 | 7.600 | | 7.549 | 0.072 | 7.074 | 7.113 | 6.700 | | 6.962 | 0.228 |
| 365 | 7.427 | 7.602 | | 7.514 | 0.124 | 7.024 | 7.053 | 6.718 | | 6.932 | 0.186 |

Note: Prism#3 (1N NaOH) and Prism#4 (Pot. Acetate) sliced at 180 day test age for SEM studies.

Table C.4 DME Data of Concrete Prisms in Modified ASTM C 1293 Tests with New Mexico Rhyolite and High Lime Fly Ash (HL3) at 25% and 35% Dosage

| NEW MEXICO 25% HIGH LIME FLY ASH (HL3) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 5.479 | 5.388 | 5.513 | 5.341 | 5.430 | 0.079 | 5.385 | 5.466 | 5.439 | 5.426 | 5.429 | 0.034 |
| 7 | 6.679 | 6.777 | 6.797 | 6.681 | 6.734 | 0.062 | 6.029 | 6.157 | 6.119 | 6.158 | 6.116 | 0.061 |
| 38 | 6.505 | 6.680 | 6.541 | 6.400 | 6.531 | 0.116 | 4.205 | 3.955 | 3.778 | 3.751 | 3.922 | 0.209 |
| 56 | 6.808 | 6.870 | 6.894 | 6.920 | 6.873 | 0.048 | 0.619 | 0.789 | 0.961 | 0.868 | 0.809 | 0.145 |
| 120 | 7.046 | 7.050 | 7.075 | 7.193 | 7.091 | 0.069 | 0.105 | 0.028 | 0.075 | Broke | 0.069 | 0.038 |
| 150 | 6.067 | 5.925 | 6.081 | 5.946 | 6.005 | 0.081 | Broke | Broke | Broke | Broke | | |
| 180 | 5.950 | 5.880 | 5.853 | 5.771 | 5.864 | 0.074 | | | | | | |
| 240 | 5.783 | 5.681 | 5.672 | | 5.712 | 0.062 | | | | | | |
| 270 | 5.584 | 5.522 | 5.380 | | 5.495 | 0.105 | | | | | | |
| 330 | 5.819 | 5.788 | 5.887 | | 5.831 | 0.051 | | | | | | |
| 365 | 5.502 | 5.389 | 5.418 | | 5.436 | 0.059 | | | | | | |

Note: Prism#4 (1N NaOH and Pot. Acetate) sliced at 180 day test age for SEM studies.

| NEW MEXICO 35% HIGH LIME FLY ASH (HL3) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 4.937 | 4.812 | 4.937 | 5.275 | 4.990 | 0.199 | 5.084 | 4.876 | 5.160 | 5.193 | 5.078 | 0.143 |
| 7 | 6.581 | 6.447 | 6.620 | 6.950 | 6.649 | 0.214 | 6.123 | 5.767 | 6.213 | 6.091 | 6.048 | 0.195 |
| 38 | 6.496 | 6.379 | 6.503 | 6.877 | 6.564 | 0.217 | 4.324 | 3.739 | 4.400 | 4.108 | 4.143 | 0.296 |
| 56 | 6.382 | 6.042 | 6.288 | 6.577 | 6.322 | 0.222 | 0.886 | 1.079 | 1.033 | 1.045 | 1.011 | 0.086 |
| 120 | 6.106 | 5.979 | 6.087 | 6.402 | 6.143 | 0.181 | 0.102 | Broke | Broke | 0.094 | 0.098 | 0.006 |
| 150 | 6.111 | 6.029 | 6.171 | 6.361 | 6.168 | 0.141 | Broke | Broke | Broke | Broke | | |
| 180 | 6.002 | 5.907 | 6.042 | 6.210 | 6.040 | 0.126 | | | | | | |
| 240 | | 5.928 | 5.877 | 6.108 | 5.971 | 0.121 | | | | | | |
| 270 | | 5.683 | 5.712 | 5.970 | 5.789 | 0.158 | | | | | | |
| 330 | | 5.593 | 5.150 | 5.559 | 5.434 | 0.247 | | | | | | |
| 365 | | 5.733 | 5.584 | 5.764 | 5.694 | 0.096 | | | | | | |

Note: Prism#1 (1N NaOH) and Prism #4 (Pot. Acetate) sliced at 180 day test age for SEM studies.

Table C.5 DME Data of Concrete Prisms in Modified ASTM C 1293 Tests with New Mexico Rhyolite and Intermediate Lime Fly Ash (IL5) at 25% and 35% Dosage

| NEW MEXICO 25% INTER. LIME FLY ASH (IL5) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 5.409 | 5.428 | 5.553 | 5.592 | 5.495 | 0.090 | 5.352 | 5.464 | 5.463 | 5.461 | 5.435 | 0.056 |
| 7 | 6.939 | 7.032 | 6.920 | 7.178 | 7.017 | 0.118 | 6.236 | 6.044 | 6.298 | 6.440 | 6.254 | 0.164 |
| 31 | 7.147 | 7.183 | 7.164 | 7.385 | 7.219 | 0.111 | 5.324 | 5.068 | 5.974 | 5.923 | 5.572 | 0.447 |
| 56 | 7.115 | 7.148 | 7.147 | 7.336 | 7.186 | 0.101 | 3.223 | 2.850 | 3.658 | 3.648 | 3.345 | 0.387 |
| 120 | 6.940 | 7.058 | 7.145 | 7.161 | 7.076 | 0.101 | 1.749 | 1.293 | 1.873 | 1.830 | 1.686 | 0.267 |
| 150 | 7.015 | 7.287 | 7.165 | 7.184 | 7.163 | 0.112 | 1.100 | 0.622 | 1.181 | 1.178 | 1.020 | 0.268 |
| 180 | 6.967 | 7.012 | 6.901 | 7.108 | 6.997 | 0.087 | 0.773 | 0.595 | 0.839 | 0.848 | 0.764 | 0.117 |
| 210 | | 6.804 | 6.724 | 6.862 | 6.797 | 0.069 | | 0.325 | 0.285 | 0.474 | 0.361 | 0.100 |
| 240 | | 6.759 | 6.680 | 6.994 | 6.811 | 0.163 | | 0.271 | 0.277 | 0.436 | 0.328 | 0.094 |
| 270 | | 6.691 | 6.589 | 7.053 | 6.778 | 0.244 | | 0.262 | 0.263 | 0.369 | 0.298 | 0.061 |
| 330 | | 6.596 | 6.553 | 7.015 | 6.721 | 0.255 | | 0.301 | 0.208 | 0.386 | 0.299 | 0.089 |
| 365 | | 6.058 | 5.739 | 6.325 | 6.041 | 0.293 | | 0.300 | 0.306 | 0.347 | 0.318 | 0.026 |

Note: Prism#1 (1N NaOH and Pot. Acetate) sliced at 180 day test age for SEM studies.

| NEW MEXICO 35% INTER. LIME FLY ASH (IL5) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 5.192 | 5.244 | 5.250 | 5.465 | 5.288 | 0.121 | 5.222 | 5.263 | 5.357 | 5.282 | 5.281 | 0.057 |
| 7 | 6.911 | 6.956 | 6.929 | 7.088 | 6.971 | 0.080 | 6.216 | 6.255 | 6.197 | 6.271 | 6.235 | 0.034 |
| 31 | 7.124 | 7.194 | 7.143 | 7.318 | 7.195 | 0.087 | 6.059 | 6.169 | 6.198 | 6.075 | 6.125 | 0.069 |
| 56 | 7.103 | 7.190 | 7.122 | 7.263 | 7.169 | 0.073 | 4.937 | 5.049 | 5.618 | 4.521 | 5.031 | 0.452 |
| 120 | 6.988 | 7.299 | 7.038 | 7.223 | 7.137 | 0.148 | 2.851 | 3.091 | 3.164 | 2.494 | 3.035 | 0.302 |
| 150 | 6.958 | 7.102 | 7.009 | 7.156 | 7.056 | 0.089 | 2.046 | 2.151 | 2.575 | 1.820 | 2.148 | 0.316 |
| 180 | 6.907 | 7.066 | 6.966 | 7.145 | 7.021 | 0.105 | 1.292 | 1.453 | 1.644 | 1.069 | 1.365 | 0.244 |
| 210 | 6.746 | 7.021 | 6.827 | | 6.865 | 0.141 | 0.801 | 0.640 | 0.880 | | 0.774 | 0.122 |
| 240 | 6.721 | 7.003 | 6.766 | | 6.830 | 0.152 | 0.669 | 0.592 | 0.751 | | 0.671 | 0.080 |
| 270 | 6.631 | 7.039 | 6.724 | | 6.798 | 0.214 | 0.499 | 0.412 | 0.533 | | 0.481 | 0.062 |
| 330 | 6.571 | 6.891 | 6.664 | | 6.709 | 0.165 | 0.511 | 0.375 | 0.519 | | 0.469 | 0.081 |
| 365 | 6.098 | 5.421 | 6.170 | | 5.896 | 0.413 | 0.398 | 0.362 | 0.465 | | 0.408 | 0.052 |

Note: Prism#4 (1N NaOH and Pot. Acetate) sliced at 180 day test age for SEM studies.

Table C.6 DME Data of Concrete Prisms in Modified ASTM C 1293 Tests with New Mexico Rhyolite and Low Lime Fly Ash (LL3) at 25% and 35% Dosage

| NEW MEXICO 25% LOW LIME FLY ASH (LL3) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 6.091 | 6.154 | 5.940 | 6.062 | 0.110 | 5.871 | 5.790 | 5.900 | 5.692 | 5.813 | 0.093 |
| 7 | 7.262 | 7.364 | 6.987 | 7.204 | 0.195 | 6.530 | 6.490 | 6.636 | 6.410 | 6.516 | 0.094 |
| 30 | 7.385 | 7.462 | 7.085 | 7.311 | 0.199 | 6.475 | 6.459 | 6.653 | 6.339 | 6.482 | 0.130 |
| 56 | 7.423 | 7.494 | 7.330 | 7.416 | 0.082 | 5.278 | 5.936 | 6.257 | 5.454 | 5.731 | 0.448 |
| 120 | 7.652 | 7.479 | 7.357 | 7.496 | 0.148 | 2.867 | 3.575 | 4.296 | 3.284 | 3.579 | 0.602 |
| 150 | 7.352 | 7.449 | 7.164 | 7.322 | 0.145 | 0.898 | 1.569 | 2.022 | 1.398 | 1.472 | 0.464 |
| 180 | 7.334 | 7.438 | 7.153 | 7.309 | 0.144 | 0.536 | 0.462 | 1.375 | 0.927 | 0.825 | 0.420 |
| 210 | 7.322 | 7.430 | 7.335 | 7.362 | 0.059 | 0.415 | 0.516 | 1.115 | 0.809 | 0.714 | 0.315 |
| 240 | 7.289 | 7.434 | 7.284 | 7.336 | 0.085 | 0.224 | 0.337 | 0.697 | 0.449 | 0.427 | 0.202 |
| 270 | 7.225 | 7.359 | 7.059 | 7.214 | 0.151 | 0.158 | 0.113 | 0.374 | 0.384 | 0.257 | 0.142 |
| 300 | 7.201 | 7.318 | 7.038 | 7.186 | 0.141 | 0.021 | 0.155 | 0.261 | | 0.146 | 0.120 |
| 330 | 7.182 | 7.293 | 7.077 | 7.184 | 0.108 | Broke | 0.177 | 0.230 | | 0.203 | 0.037 |
| 365 | 7.131 | 7.269 | 7.215 | 7.205 | 0.070 | Broke | 0.191 | Broke | | 0.191 | |

| NEW MEXICO 35% LOW LIMEFLY ASH (LL3) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | Potassium Acetate (DME in psi x 10^6) | | | | |
| | #1 | #2 | #3 | Avg. | Std Dev. | #1 | #2 | #3 | Avg. | Std Dev. |
| 0 | 6.583 | 5.579 | 5.348 | 5.837 | 0.657 | 5.537 | 5.496 | 5.327 | 5.453 | 0.112 |
| 7 | 7.110 | 7.331 | 7.032 | 7.157 | 0.155 | 6.415 | 6.415 | 6.168 | 6.332 | 0.142 |
| 30 | 7.198 | 7.440 | 7.122 | 7.253 | 0.166 | 6.473 | 6.437 | 6.164 | 6.358 | 0.169 |
| 56 | 7.238 | 7.335 | 7.153 | 7.242 | 0.091 | 6.199 | 6.344 | 5.968 | 6.171 | 0.190 |
| 120 | 7.397 | 7.499 | 7.147 | 7.347 | 0.181 | 5.304 | 5.647 | 5.435 | 5.462 | 0.173 |
| 150 | 7.470 | 7.307 | 7.134 | 7.304 | 0.168 | 3.499 | 3.856 | 3.326 | 3.560 | 0.270 |
| 180 | 7.267 | 7.414 | 7.129 | 7.270 | 0.143 | 2.639 | 3.072 | 2.558 | 2.757 | 0.276 |
| 210 | 7.288 | 7.410 | 7.130 | 7.276 | 0.141 | 2.082 | 2.173 | 1.951 | 2.069 | 0.112 |
| 240 | 7.208 | 7.260 | 7.101 | 7.190 | 0.081 | 1.441 | 1.574 | 1.283 | 1.432 | 0.146 |
| 270 | 7.152 | 7.353 | 7.053 | 7.186 | 0.153 | 0.909 | 0.968 | 0.858 | 0.912 | 0.055 |
| 300 | 7.308 | 7.378 | 7.054 | 7.247 | 0.171 | 0.624 | 0.713 | 0.634 | 0.657 | 0.049 |
| 330 | 7.291 | 7.362 | 7.045 | 7.233 | 0.166 | 0.455 | 0.635 | 0.425 | 0.505 | 0.114 |
| 365 | 7.236 | 7.176 | 7.020 | 7.144 | 0.112 | 0.235 | 0.488 | 0.154 | 0.293 | 0.174 |

Table C.7 DME Data of Concrete Prisms in Modified ASTM C 1293 Tests with Spratt Limestone and Slag at 40% and 50% Dosage

| SPRATT LIMESTONE 40% SLAG | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 5.391 | 5.206 | 5.341 | 5.089 | 5.257 | 0.136 | 5.278 | 5.403 | 5.261 | 5.395 | 5.334 | 0.075 |
| 28 | 7.471 | 7.279 | 7.449 | 7.234 | 7.358 | 0.119 | 6.709 | 6.903 | 6.714 | 6.702 | 6.757 | 0.097 |
| 56 | 7.544 | 7.351 | 7.518 | 7.316 | 7.432 | 0.115 | 6.689 | 6.928 | 6.773 | 6.749 | 6.785 | 0.102 |
| 90 | 7.558 | 7.399 | 7.571 | 7.337 | 7.466 | 0.117 | 6.761 | 6.937 | 6.802 | 6.808 | 6.827 | 0.076 |
| 120 | 7.583 | 7.413 | 7.592 | 7.372 | 7.490 | 0.114 | 6.750 | 6.968 | 6.807 | 6.771 | 6.824 | 0.099 |
| 180 | 7.568 | 7.406 | 7.575 | 7.346 | 7.474 | 0.115 | 6.666 | 6.872 | 6.754 | 6.776 | 6.767 | 0.085 |
| 210 | 7.523 | 7.361 | 7.528 | | 7.471 | 0.095 | 6.664 | 6.841 | 6.719 | | 6.742 | 0.091 |
| 240 | 7.535 | 7.341 | 7.528 | | 7.468 | 0.110 | 6.588 | 6.763 | 6.634 | | 6.662 | 0.091 |
| 270 | 7.422 | 7.264 | 7.466 | | 7.384 | 0.106 | 6.496 | 6.770 | 6.552 | | 6.606 | 0.145 |
| 300 | 7.440 | 7.232 | 7.414 | | 7.362 | 0.113 | 6.442 | 6.677 | 6.496 | | 6.538 | 0.123 |
| 365 | | | | | | | | | | | | |

Note: Prism#4 (1N NaOH and Pot. Acetate) sliced at 180 day test age for SEM studies.

| SPRATT LIMESTONE 50% SLAG | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 5.411 | 5.004 | 5.081 | 5.130 | 5.156 | 0.177 | 4.988 | 5.300 | 5.094 | 5.061 | 5.111 | 0.133 |
| 28 | 7.405 | 7.270 | 7.307 | 7.323 | 7.326 | 0.057 | 6.580 | 6.911 | 6.748 | 6.700 | 6.735 | 0.137 |
| 56 | 7.497 | 7.357 | 7.390 | 7.397 | 7.410 | 0.060 | 6.656 | 6.966 | 6.801 | 6.752 | 6.794 | 0.130 |
| 90 | 7.541 | 7.408 | 7.434 | 7.443 | 7.457 | 0.058 | 6.683 | 6.979 | 6.801 | 6.757 | 6.805 | 0.126 |
| 120 | 7.282 | 7.253 | 7.399 | 7.306 | 7.310 | 0.063 | 6.785 | 6.957 | 6.771 | 6.657 | 6.792 | 0.124 |
| 180 | 7.521 | 7.462 | 7.464 | 7.495 | 7.485 | 0.028 | 6.764 | 6.949 | 6.714 | 6.679 | 6.777 | 0.120 |
| 210 | 7.622 | 7.459 | 7.464 | | 7.515 | 0.093 | 6.646 | 6.942 | 6.664 | | 6.751 | 0.166 |
| 240 | 7.600 | 7.476 | 7.476 | | 7.518 | 0.071 | 6.612 | 6.903 | 6.610 | | 6.708 | 0.169 |
| 270 | 7.537 | 7.439 | 7.447 | | 7.474 | 0.055 | 6.579 | 6.606 | 6.530 | | 6.572 | 0.039 |
| 300 | 7.522 | 7.420 | 7.432 | | 7.458 | 0.056 | 6.534 | 6.823 | 6.484 | | 6.614 | 0.183 |
| | | | | | | | | | | | | |

Note: Prism#4 (1N NaOH and Pot. Acetate) sliced at 180 day test age for SEM studies.

Table C.8 DME Data of Concrete Prisms in Modified ASTM C 1293 Tests with NM Rhyolite and Slag at 40% and 50% Dosage

| NEW MEXICO 40% SLAG | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 5.058 | 5.013 | 5.285 | 5.269 | 5.156 | 0.141 | 5.416 | 5.221 | 5.195 | 5.386 | 5.304 | 0.112 |
| 7 | 6.472 | 6.607 | 6.854 | 6.877 | 6.702 | 0.196 | 6.533 | 6.366 | 6.310 | 6.520 | 6.432 | 0.111 |
| 28 | 6.963 | 6.881 | 7.137 | 7.140 | 7.030 | 0.129 | 6.537 | 6.451 | 6.383 | 6.610 | 6.495 | 0.099 |
| 56 | 6.841 | 6.922 | 7.180 | 7.155 | 7.025 | 0.169 | 6.329 | 6.118 | 6.002 | 6.340 | 6.197 | 0.165 |
| 90 | 6.826 | 6.905 | 7.180 | 7.167 | 7.019 | 0.181 | 3.597 | 4.594 | 3.678 | 4.665 | 4.134 | 0.574 |
| 120 | 6.987 | 6.872 | 7.140 | 7.140 | 7.035 | 0.130 | 2.128 | 3.031 | 2.265 | 2.940 | 2.591 | 0.461 |
| 180 | 6.973 | 6.853 | 7.058 | 7.072 | 6.989 | 0.100 | 1.194 | 1.854 | 0.962 | 1.437 | 1.362 | 0.381 |
| 210 | | 6.772 | 7.087 | 6.970 | 6.929 | 0.159 | | 0.822 | 0.423 | 0.677 | 0.641 | 0.202 |
| 240 | | 6.572 | 6.972 | 6.768 | 6.772 | 0.200 | | 0.429 | 0.442 | 0.409 | 0.427 | 0.016 |
| 270 | | 6.543 | 6.972 | 6.706 | 6.758 | 0.217 | | 0.405 | 0.366 | 0.257 | 0.343 | 0.077 |
| 300 | | 6.481 | 6.929 | 6.619 | 6.705 | 0.229 | | 0.388 | 0.369 | 0.227 | 0.328 | 0.088 |
| 330 | | 6.383 | 6.878 | 6.541 | 6.631 | 0.253 | | 0.418 | 0.323 | 0.361 | 0.367 | 0.048 |
| 365 | | 6.337 | 6.766 | 6.562 | 6.552 | 0.215 | | 0.450 | 0.428 | 0.320 | 0.399 | 0.070 |

Note: Prism#1 (1N NaOH and Pot. Acetate) sliced at 180 day test age for SEM studies.

| NEW MEXICO 50% SLAG | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 5.234 | 5.083 | 5.177 | 5.136 | 5.158 | 0.064 | 5.001 | 5.214 | 5.130 | 5.045 | 5.097 | 0.094 |
| 7 | 7.043 | 6.895 | 7.062 | 6.958 | 6.990 | 0.078 | 6.392 | 6.501 | 6.403 | 6.343 | 6.410 | 0.066 |
| 28 | 7.321 | 7.161 | 7.331 | 7.217 | 7.258 | 0.082 | 6.527 | 6.665 | 6.561 | 6.498 | 6.563 | 0.073 |
| 56 | 7.362 | 7.240 | 7.413 | 7.303 | 7.330 | 0.074 | 6.520 | 6.677 | 6.500 | 6.460 | 6.539 | 0.095 |
| 90 | 7.349 | 7.254 | 7.408 | 7.311 | 7.330 | 0.065 | 6.452 | 6.757 | 6.509 | 6.680 | 6.600 | 0.143 |
| 120 | 7.602 | 7.453 | 7.465 | 7.486 | 7.502 | 0.068 | 6.684 | 6.986 | 6.779 | 6.748 | 6.799 | 0.131 |
| 180 | 7.359 | 7.264 | 7.414 | 7.309 | 7.337 | 0.065 | 3.582 | 3.290 | 3.165 | 3.264 | 3.325 | 0.179 |
| 210 | 7.309 | 7.269 | 7.352 | | 7.310 | 0.042 | | 2.693 | 2.260 | 2.038 | 2.331 | 0.333 |
| 240 | 7.214 | 7.211 | 7.318 | | 7.248 | 0.061 | | 2.144 | 1.909 | 1.692 | 1.915 | 0.226 |
| 270 | 7.232 | 7.208 | 7.224 | | 7.221 | 0.012 | | 2.087 | 1.551 | 1.399 | 1.679 | 0.361 |
| 300 | 7.251 | 7.180 | 7.271 | | 7.234 | 0.048 | | 1.922 | 1.241 | 1.068 | 1.410 | 0.452 |
| 330 | 7.214 | 7.147 | 7.263 | | 7.208 | 0.058 | | 1.909 | 1.137 | 0.791 | 1.279 | 0.572 |
| 365 | 7.153 | 7.140 | 7.075 | | 7.122 | 0.042 | | 1.376 | 0.802 | 0.787 | 0.988 | 0.336 |

Note: Prism#4 (1N NaOH) and Prism#1 (Pot. Acetate) sliced at 180 day test age for SEM studies.

Table C.9 DME Data of Concrete Prisms in Modified ASTM C 1293 Tests with Illinois Dolomite and Slag at 40% Dosage

| ILLINOIS DOLOMITE 40% SLAG | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 4.651 | 4.895 | 5.311 | 4.918 | 4.944 | 0.273 | 4.961 | 4.948 | 5.161 | 4.645 | 4.929 | 0.213 |
| 7 | 6.956 | 7.178 | 7.390 | 7.067 | 7.148 | 0.185 | 6.564 | 6.716 | 6.734 | 5.743 | 6.439 | 0.471 |
| 28 | 7.343 | 7.539 | 7.788 | 7.456 | 7.531 | 0.189 | 6.843 | 6.940 | 7.002 | 6.454 | 6.810 | 0.246 |
| 56 | 7.476 | 7.644 | 7.903 | 7.573 | 7.649 | 0.183 | 6.941 | 7.056 | 7.116 | 6.657 | 6.942 | 0.203 |
| 90 | 7.510 | 7.683 | 7.942 | 7.608 | 7.686 | 0.185 | 6.982 | 7.083 | 7.147 | 6.691 | 6.976 | 0.201 |
| 120 | 7.553 | 7.709 | 7.982 | 7.631 | 7.719 | 0.187 | 7.017 | 7.106 | 7.173 | 6.718 | 7.003 | 0.201 |
| 150 | 7.574 | 7.740 | 8.017 | 7.652 | 7.746 | 0.193 | 7.045 | 7.130 | 7.194 | 6.749 | 7.030 | 0.196 |
| 180 | 7.616 | 7.778 | 8.054 | 7.696 | 7.786 | 0.190 | 7.067 | 7.198 | 7.197 | 6.776 | 7.060 | 0.199 |
| 210 | 7.648 | 7.809 | 8.078 | | 7.845 | 0.217 | 7.098 | 7.361 | 7.217 | | 7.225 | 0.131 |
| 240 | 7.679 | 7.835 | 8.117 | | 7.877 | 0.222 | 7.088 | 7.236 | 7.204 | | 7.176 | 0.078 |
| 270 | 7.676 | 7.750 | 8.122 | | 7.849 | 0.239 | 7.367 | 7.229 | 7.182 | | 7.260 | 0.096 |
| 300 | 7.723 | 7.812 | 8.149 | | 7.895 | 0.225 | 7.501 | 7.144 | 7.149 | | 7.265 | 0.205 |
| 330 | 7.691 | 7.768 | 8.104 | | 7.855 | 0.219 | 7.537 | 7.203 | 7.151 | | 7.297 | 0.209 |
| 365 | 7.733 | 7.846 | 8.142 | | 7.907 | 0.211 | 7.543 | 7.253 | 7.142 | | 7.313 | 0.207 |

Note: Prism#4 (1N NaOH and Pot. Acetate) sliced at 180 day test age for SEM studies.

Table C.10 DME Data of Mortar Bars in Standard and Modified ASTM C 1567 Tests with Spratt Limestone and Selected Fly Ashes at 25% Dosage

| SPRATT 25% HIGH LIME FLY ASH (HL3) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 7 | 4.006 | 3.820 | 3.782 | 3.825 | 3.858 | 0.100 | 2.658 | 2.782 | 2.785 | 2.609 | 2.709 | 0.089 |
| 11 | 3.894 | 3.790 | 3.722 | 3.791 | 3.799 | 0.071 | 2.109 | 2.266 | 2.413 | 2.186 | 2.244 | 0.130 |
| 14 | 3.590 | 3.469 | 3.466 | 3.485 | 3.503 | 0.059 | 1.978 | 2.149 | 2.271 | 2.140 | 2.134 | 0.120 |
| 21 | 2.645 | 2.442 | 2.624 | | 2.570 | 0.111 | 1.616 | 1.726 | 1.939 | | 1.760 | 0.164 |
| 28 | 1.952 | 1.758 | 1.751 | | 1.820 | 0.114 | 1.259 | 1.336 | 1.594 | | 1.397 | 0.175 |
| 42 | 1.660 | 1.504 | 1.343 | | 1.502 | 0.159 | 1.051 | 1.095 | 1.348 | | 1.165 | 0.160 |
| 56 | 1.526 | 1.407 | 1.265 | | 1.399 | 0.131 | 0.962 | 0.981 | 1.291 | | 1.078 | 0.185 |
| SPRATT 25% INTERMEDIATE LIME FLY ASH (IL5) | | | | | | | | | | | | |
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 7 | 4.215 | 4.296 | 4.339 | | 4.284 | 0.063 | 4.180 | 4.092 | 4.166 | 4.137 | 4.144 | 0.039 |
| 11 | 4.238 | 4.256 | 4.286 | | 4.260 | 0.024 | 4.086 | 4.110 | 4.192 | 4.137 | 4.131 | 0.046 |
| 14 | 4.076 | 4.178 | 4.151 | | 4.135 | 0.053 | 4.021 | 4.087 | 4.141 | 4.152 | 4.100 | 0.060 |
| 21 | 3.850 | 3.933 | 3.951 | | 3.911 | 0.054 | 4.033 | 3.981 | 4.046 | | 4.020 | 0.034 |
| 28 | 3.383 | 3.461 | 3.471 | | 3.438 | 0.048 | 3.962 | 3.960 | 4.008 | | 3.977 | 0.027 |
| 42 | 3.142 | 3.117 | 3.199 | | 3.153 | 0.042 | 3.955 | 3.859 | 3.989 | | 3.934 | 0.068 |
| 56 | 2.646 | 2.797 | 2.752 | | 2.732 | 0.078 | 3.832 | 3.695 | 3.780 | | 3.769 | 0.069 |
| SPRATT 25% LOW LIME FLY ASH (LL3) | | | | | | | | | | | | |
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 7 | 4.201 | 4.245 | 4.439 | 4.273 | 4.290 | 0.104 | 4.123 | 4.109 | 3.999 | 4.116 | 4.087 | 0.059 |
| 11 | 4.165 | 4.280 | 4.403 | 4.239 | 4.272 | 0.099 | 4.028 | 4.036 | 3.994 | 4.119 | 4.044 | 0.053 |
| 14 | 4.042 | 4.099 | 4.272 | 4.177 | 4.147 | 0.100 | 4.041 | 4.011 | 3.963 | 4.099 | 4.029 | 0.057 |
| 21 | 3.810 | 3.896 | 4.062 | | 3.923 | 0.128 | 3.850 | 3.884 | 3.807 | | 3.847 | 0.038 |
| 28 | 3.461 | 3.525 | 3.671 | | 3.552 | 0.108 | 3.791 | 3.820 | 3.756 | | 3.789 | 0.032 |
| 42 | 3.184 | 3.284 | 3.436 | | 3.301 | 0.127 | 3.764 | 3.791 | 3.701 | | 3.752 | 0.046 |
| 56 | 2.939 | 3.002 | 3.131 | | 3.024 | 0.098 | 3.726 | 3.764 | 3.662 | | 3.717 | 0.052 |

Note: DME for day '0' and day '3' not available due to equipment breakdown at those days

Table C.11 DME Data of Mortar Bars in Standard and Modified ASTM C 1567 Tests with New Mexico Rhyolite and High Lime Fly Ash at 25% Dosage

| NEW MEXICO 25% HIGH LIME FLY ASH (HL3) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 3.794 | 4.071 | 3.884 | 3.995 | 3.936 | 0.122 | 4.170 | 4.119 | 4.107 | 4.004 | 4.100 | 0.070 |
| 3 | 2.326 | 2.442 | 2.449 | 2.476 | 2.423 | 0.067 | 1.948 | 1.728 | 0.818 | 2.465 | 1.740 | 0.688 |
| 7 | 1.923 | 2.186 | 2.112 | 2.275 | 2.124 | 0.150 | broke | 1.352 | 1.776 | 1.678 | 1.602 | 0.222 |
| 11 | 1.717 | 1.925 | 1.888 | 1.983 | 1.878 | 0.114 | | 0.976 | 0.612 | 0.538 | 0.709 | 0.235 |
| 14 | 1.552 | 1.698 | 1.743 | 1.820 | 1.703 | 0.113 | | 0.597 | 0.760 | 0.609 | 0.655 | 0.091 |
| 21 | 1.336 | 1.471 | 1.500 | | 1.436 | 0.088 | | 0.759 | 0.799 | 0.672 | 0.743 | 0.065 |
| 28 | 1.235 | 1.332 | 1.344 | | 1.304 | 0.060 | | 0.721 | 0.654 | 0.739 | 0.705 | 0.045 |
| 42 | 1.185 | 1.344 | 1.320 | | 1.283 | 0.086 | | 0.527 | 1.981 | 0.701 | 1.070 | 0.794 |
| 56 | 1.184 | 1.314 | 1.264 | | 1.254 | 0.066 | | 0.880 | 1.203 | 2.107 | 1.397 | 0.636 |

Table C.12 DME Data of Mortar Bars in Standard and Modified ASTM C 1567 Tests with New Mexico Rhyolite and Intermediate and Low Lime Fly Ash at 25% Dosage

| NEW MEXICO 25% INTERMEDIATE LIME FLY ASH (IL5) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | Potassium Acetate (DME in psi x 10^6) | | | | |
| | #1 | #2 | #3 | Avg. | Std Dev. | #1 | #2 | #3 | Avg. | Std Dev. |
| 0 | 4.635 | 4.356 | 4.241 | 4.410 | 0.203 | 4.467 | 4.383 | 4.402 | 4.417 | 0.044 |
| 3 | 4.648 | 4.218 | 4.109 | 4.325 | 0.285 | 4.373 | 4.362 | 4.453 | 4.396 | 0.050 |
| 7 | 3.610 | 3.431 | 3.917 | 3.652 | 0.246 | 4.199 | 4.206 | 4.191 | 4.199 | 0.007 |
| 11 | 3.348 | 3.165 | 3.294 | 3.269 | 0.094 | 0.864 | 3.376 | 0.841 | 1.693 | 1.457 |
| 14 | 3.057 | 2.991 | 3.152 | 3.066 | 0.081 | 0.553 | 2.603 | 0.781 | 1.312 | 1.123 |
| 21 | 2.681 | 2.589 | 2.832 | 2.701 | 0.123 | 0.599 | 0.625 | 0.684 | 0.636 | 0.043 |
| 28 | 2.387 | 2.304 | 2.601 | 2.431 | 0.153 | 2.274 | 0.661 | 0.838 | 1.258 | 0.884 |
| 42 | 1.905 | 1.867 | 2.185 | 1.986 | 0.173 | 0.893 | 1.005 | 0.954 | 0.951 | 0.056 |
| 56 | 1.675 | 1.637 | 1.850 | 1.721 | 0.113 | 0.857 | 1.130 | 1.303 | 1.097 | 0.225 |
| NEW MEXICO 25% LOW LIME FLY ASH (LL3) | | | | | | | | | | |
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | Potassium Acetate (DME in psi x 10^6) | | | | |
| | #1 | #2 | #3 | Avg. | Std Dev. | #1 | #2 | #3 | Avg. | Std Dev. |
| 0 | 4.412 | 4.351 | 4.241 | 4.335 | 0.087 | 3.995 | 4.478 | 4.463 | 4.312 | 0.274 |
| 3 | 4.211 | 4.188 | 4.109 | 4.169 | 0.054 | 4.055 | 4.295 | 4.384 | 4.245 | 0.170 |
| 7 | 4.008 | 3.923 | 3.917 | 3.949 | 0.051 | 4.040 | 4.267 | 4.313 | 4.206 | 0.146 |
| 11 | 3.592 | 3.502 | 3.294 | 3.463 | 0.153 | 3.950 | 4.169 | 4.103 | 4.074 | 0.112 |
| 14 | 3.317 | 3.263 | 3.152 | 3.244 | 0.084 | 3.960 | 4.064 | 4.095 | 4.040 | 0.071 |
| 21 | 2.991 | 2.941 | 2.832 | 2.922 | 0.081 | | 1.864 | 2.823 | 2.343 | 0.678 |
| 28 | 2.731 | 2.694 | 2.601 | 2.675 | 0.067 | | 1.202 | 1.631 | 1.417 | 0.304 |
| 42 | 2.260 | 2.266 | 2.185 | 2.237 | 0.045 | | 1.037 | 1.651 | 1.344 | 0.434 |
| 56 | 1.946 | 1.929 | 1.850 | 1.908 | 0.051 | | 1.683 | 1.573 | 1.628 | 0.078 |

Table C.13 DME Data of Mortar Bars in Standard and Modified ASTM C 1567 Tests with North Carolina Argillite and Selected Fly Ashes at 25% Dosage

| NORTH CAROLINA 25% HIGH LIME FLY ASH (HL3) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 3.966 | 4.182 | 4.109 | 4.196 | 4.113 | 0.105 | 4.081 | 4.065 | 4.140 | 4.166 | 4.113 | 0.048 |
| 3 | 3.264 | 3.378 | 3.415 | 3.388 | 3.361 | 0.067 | 3.856 | 3.802 | 3.898 | 3.885 | 3.860 | 0.043 |
| 7 | 3.184 | 3.276 | 3.305 | 3.330 | 3.274 | 0.063 | 1.572 | 1.407 | 1.548 | 1.649 | 1.544 | 0.101 |
| 11 | 3.125 | 3.153 | 3.213 | 3.256 | 3.186 | 0.059 | 1.693 | 1.546 | 1.574 | 1.570 | 1.596 | 0.066 |
| 14 | 2.927 | 2.952 | 2.998 | 3.005 | 2.970 | 0.037 | 1.829 | 1.705 | 1.897 | 1.890 | 1.830 | 0.089 |
| 21 | 2.811 | 2.738 | 2.944 | | 2.831 | 0.105 | 1.870 | 1.708 | 1.853 | | 1.810 | 0.089 |
| 28 | 2.758 | 2.790 | 2.858 | | 2.802 | 0.051 | 2.000 | 1.777 | 1.984 | | 1.920 | 0.124 |
| 42 | 2.674 | 2.696 | 2.785 | | 2.718 | 0.059 | 2.082 | 1.928 | 2.078 | | 2.029 | 0.088 |
| 56 | 2.659 | 2.703 | 2.758 | | 2.706 | 0.050 | 2.202 | 2.006 | 2.123 | | 2.111 | 0.098 |
| NORTH CAROLINA 25% INTERMEDIATE LIME FLY ASH (IL5) | | | | | | | | | | | | |
| Age (Days) | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 4.143 | 4.322 | 4.181 | | 4.215 | 0.094 | 3.669 | 4.054 | 4.136 | 4.229 | 4.022 | 0.246 |
| 3 | 4.249 | 4.391 | 4.209 | | 4.283 | 0.095 | 4.431 | 4.158 | 4.244 | 4.270 | 4.276 | 0.114 |
| 7 | 3.976 | 4.148 | 3.954 | | 4.026 | 0.106 | 4.302 | 4.112 | 4.213 | 4.216 | 4.210 | 0.078 |
| 11 | 3.825 | 4.059 | 3.796 | | 3.893 | 0.144 | 4.292 | 4.107 | 4.224 | 4.203 | 4.206 | 0.076 |
| 14 | 3.564 | 3.681 | 3.515 | | 3.586 | 0.085 | 4.282 | 4.090 | 4.214 | 4.178 | 4.191 | 0.080 |
| 21 | 3.450 | 3.568 | 3.430 | | 3.483 | 0.075 | 4.256 | 4.095 | 4.188 | | 4.179 | 0.081 |
| 28 | 3.273 | 3.414 | 3.276 | | 3.321 | 0.080 | 4.287 | 4.102 | 4.219 | | 4.203 | 0.094 |
| 42 | 3.050 | 3.163 | 3.037 | | 3.083 | 0.070 | 4.344 | 4.142 | 4.260 | | 4.249 | 0.101 |
| 56 | 2.867 | 2.963 | 2.866 | | 2.899 | 0.056 | 4.357 | 4.162 | 4.272 | | 4.264 | 0.098 |
| NORTH CAROLINA 25% LOW LIME FLY ASH (LL3) | | | | | | | | | | | | |
| Age (Days) | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 4.529 | 4.312 | 4.332 | 4.416 | 4.397 | 0.099 | 4.372 | 4.410 | 4.364 | 4.431 | 4.394 | 0.032 |
| 3 | 4.579 | 4.423 | 4.436 | 4.508 | 4.487 | 0.072 | 4.361 | 4.369 | 4.350 | 4.419 | 4.375 | 0.031 |
| 7 | 4.340 | 4.198 | 4.171 | 4.245 | 4.238 | 0.074 | 4.276 | 4.219 | 4.287 | 4.349 | 4.283 | 0.053 |
| 11 | 4.169 | 4.069 | 4.016 | 4.110 | 4.091 | 0.065 | 4.246 | 4.179 | 4.262 | 4.323 | 4.252 | 0.059 |
| 14 | 3.941 | 3.815 | 3.782 | 3.869 | 3.852 | 0.069 | 4.185 | 4.106 | 4.170 | 4.231 | 4.173 | 0.051 |
| 21 | 3.876 | 3.737 | 3.716 | | 3.776 | 0.087 | 4.151 | 4.068 | 4.129 | | 4.116 | 0.043 |
| 28 | 3.734 | 3.591 | 3.570 | | 3.631 | 0.089 | 4.131 | 4.060 | 4.124 | | 4.105 | 0.039 |
| 42 | 3.541 | 3.376 | 3.373 | | 3.430 | 0.096 | 4.135 | 4.072 | 4.152 | | 4.120 | 0.042 |
| 56 | 3.349 | 3.219 | 3.193 | | 3.254 | 0.084 | 4.171 | 4.072 | 4.172 | | 4.138 | 0.057 |

Table C.14 DME Data of Mortar Bars in Standard and Modified ASTM C 1567 Tests with South Dakota Quartzite and Selected Fly Ashes at 25% Dosage

| SOUTH DAKOTA 25% HIGH LIME FLY ASH (HL3) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Age (Days) | 1N NaOH (DME in psi x 10^6) | | | | | | Potassium Acetate (DME in psi x 10^6) | | | | | |
| | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 4.369 | 4.446 | 4.328 | 4.367 | 4.377 | 0.049 | 4.296 | 4.265 | 4.440 | 4.448 | 4.362 | 0.095 |
| 3 | 3.395 | 3.392 | 3.416 | 3.409 | 3.403 | 0.011 | 3.008 | 2.975 | 3.065 | 3.092 | 3.035 | 0.053 |
| 7 | 3.047 | 3.101 | 3.163 | 3.136 | 3.112 | 0.050 | 2.313 | 2.330 | 2.430 | 2.360 | 2.358 | 0.052 |
| 11 | 3.014 | 3.033 | 3.127 | 3.089 | 3.066 | 0.052 | 2.117 | 2.087 | 2.102 | 2.153 | 2.115 | 0.028 |
| 14 | 2.928 | 3.001 | 3.020 | 2.990 | 2.985 | 0.040 | 2.220 | 2.156 | 2.154 | 2.185 | 2.179 | 0.031 |
| 21 | 2.747 | 2.813 | 2.826 | | 2.795 | 0.043 | 2.237 | 2.313 | 2.309 | | 2.286 | 0.043 |
| 28 | 2.611 | 2.674 | 2.692 | | 2.659 | 0.042 | 2.579 | 2.446 | 2.408 | | 2.478 | 0.090 |
| 42 | 2.483 | 2.498 | 2.536 | | 2.506 | 0.027 | 2.817 | 2.624 | 2.686 | | 2.709 | 0.099 |
| 56 | 2.390 | 2.406 | 2.436 | | 2.411 | 0.023 | 2.945 | 2.782 | 2.866 | | 2.864 | 0.081 |
| SOUTH DAKOTA 25% INTERMEDIATE LIME FLY ASH (IL3) | | | | | | | | | | | | |
| Age (Days) | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 4.484 | 4.493 | 4.604 | 4.489 | 4.518 | 0.058 | 4.499 | 4.494 | 4.462 | 4.683 | 4.535 | 0.100 |
| 3 | 4.500 | 4.541 | 4.603 | 4.881 | 4.631 | 0.172 | 4.694 | 4.650 | 4.656 | 4.734 | 4.684 | 0.039 |
| 7 | 4.013 | 3.986 | 4.017 | 4.022 | 4.010 | 0.016 | 4.540 | 4.634 | 4.472 | 4.712 | 4.590 | 0.105 |
| 11 | 3.687 | 3.643 | 3.668 | 3.834 | 3.708 | 0.086 | 4.503 | 4.532 | 4.399 | 4.491 | 4.481 | 0.057 |
| 14 | 3.449 | 3.387 | 3.421 | 3.529 | 3.446 | 0.061 | 4.479 | 4.516 | 4.388 | 4.416 | 4.450 | 0.058 |
| 21 | 3.150 | 3.072 | 3.106 | | 3.109 | 0.039 | 4.537 | 4.551 | 4.391 | | 4.493 | 0.089 |
| 28 | 2.955 | 2.870 | 2.920 | | 2.915 | 0.042 | 4.530 | 4.591 | 4.491 | | 4.537 | 0.050 |
| 42 | 2.674 | 2.572 | 2.613 | | 2.619 | 0.051 | 4.599 | 4.629 | 4.539 | | 4.589 | 0.046 |
| 56 | 2.517 | 2.409 | 2.435 | | 2.453 | 0.056 | 4.640 | 4.653 | 4.544 | | 4.612 | 0.060 |
| SOUTH DAKOTA 25% LOW LIME FLY ASH (LL3) | | | | | | | | | | | | |
| Age (Days) | #1 | #2 | #3 | #4 | Avg. | Std Dev. | #1 | #2 | #3 | #4 | Avg. | Std Dev. |
| 0 | 4.721 | 4.914 | 4.505 | 4.817 | 4.739 | 0.175 | 4.721 | 4.471 | 4.864 | 4.640 | 4.674 | 0.164 |
| 3 | 4.724 | 4.825 | 4.621 | 4.886 | 4.764 | 0.116 | 4.674 | 4.489 | 4.732 | 4.545 | 4.610 | 0.112 |
| 7 | 4.278 | 4.425 | 4.184 | 4.306 | 4.298 | 0.100 | 4.590 | 4.383 | 4.708 | 4.559 | 4.560 | 0.134 |
| 11 | 3.981 | 4.166 | 3.906 | 4.052 | 4.026 | 0.111 | 4.564 | 4.337 | 4.435 | 4.543 | 4.470 | 0.105 |
| 14 | 3.724 | 3.894 | 3.669 | 3.851 | 3.784 | 0.106 | 4.535 | 4.293 | 4.411 | 4.527 | 4.441 | 0.114 |
| 21 | 3.381 | 3.568 | 3.338 | | 3.429 | 0.122 | 4.473 | 4.288 | 4.393 | | 4.385 | 0.093 |
| 28 | 3.125 | 3.345 | 3.085 | | 3.185 | 0.140 | 4.459 | 4.273 | 4.394 | | 4.375 | 0.094 |
| 42 | 2.825 | 3.004 | 2.788 | | 2.873 | 0.115 | 4.468 | 4.272 | 4.401 | | 4.380 | 0.099 |
| 56 | 2.624 | 2.797 | 2.582 | | 2.667 | 0.114 | 4.479 | 4.316 | 4.425 | | 4.407 | 0.083 |

## APPENDIX-D INDUCTIVELY COUPLED PLASMA (ICP) TEST DATA

Table D.1 Concentration of Selected Elements in the Silica Dissolution Study at Room Temperature Using ICP Method

| Element Concentration Detected | Fused Silica Exposure Combination | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | NaOH+Si | | NaOH+Si+CH | | KAc+Si | | KAc+Si+CH | |
| Si | hrs | ppm | hrs | ppm | hrs | ppm | hrs | ppm |
| | 8.5 | 605.50 | 26 | 387.50 | 8.5 | 167.50 | 8.5 | 7.00 |
| | 26 | 560.00 | 48 | 473.50 | 26 | 183.00 | 26 | 0.00 |
| | 48 | 846.00 | 168 | 600.50 | 168 | 160.50 | 168 | 29.50 |
| | 168 | 666.00 | 384 | 571.00 | 336 | 97.00 | 336 | 2.00 |
| | 384 | 1338.00 | 672 | 633.00 | 504 | 126.00 | 504 | 38.00 |
| | 672 | 1534.00 | | | 672 | 163.00 | 672 | 22.50 |
| Ca | hrs | ppm | hrs | ppm | hrs | ppm | hrs | ppm |
| | 8.5 | 45.00 | 26 | 15.50 | 8.5 | 3.50 | 8.5 | 278.00 |
| | 26 | 36.00 | 48 | 45.00 | 26 | 5.50 | 26 | 232.00 |
| | 48 | 56.00 | 168 | 29.50 | 168 | 0.50 | 168 | 672.50 |
| | 168 | 4.00 | 384 | 59.50 | 336 | 48.00 | 336 | 75.00 |
| | 384 | 20.50 | 672 | 65.00 | 504 | 0.00 | 504 | 652.00 |
| | 672 | -7.50 | | | 672 | -2.50 | 672 | 1001.00 |
| Na | hrs | ppm | hrs | ppm | hrs | ppm | hrs | ppm |
| | 8.5 | 7756.00 | 26 | 7478.00 | 8.5 | 241.00 | 8.5 | 345.00 |
| | 26 | 7571.50 | 48 | 7875.00 | 26 | 235.50 | 26 | 305.00 |
| | 48 | 7442.50 | 168 | 7621.00 | 168 | 231.50 | 168 | 281.00 |
| | 168 | 7430.00 | 384 | 7749.00 | 336 | 314.00 | 336 | 247.00 |
| | 384 | 7818.50 | 672 | 6529.50 | 504 | 200.50 | 504 | 215.50 |
| | 672 | 7533.50 | | | 672 | 224.00 | 672 | 230.50 |
| K | hrs | ppm | hrs | ppm | hrs | ppm | hrs | ppm |
| | 8.5 | 83.50 | 26 | 90.00 | 8.5 | 123173.50 | 8.5 | 132103.50 |
| | 26 | 29.00 | 48 | 83.50 | 26 | 122917.00 | 26 | 127462.50 |
| | 48 | 39.50 | 168 | 54.50 | 168 | 122640.50 | 168 | 123948.00 |
| | 168 | 40.00 | 384 | 84.00 | 336 | 123351.00 | 336 | 120511.00 |
| | 384 | 38.00 | 672 | 44.00 | 504 | 119272.50 | 504 | 117170.00 |
| | 672 | 38.00 | | | 672 | 121118.50 | 672 | 122007.50 |

*Note: All concentrations are in 'ppm'- parts per million. Si- Fused Silica, CH-Calcium Hydroxide (Lime), KAc- 50% wt. potassium acetate deicer solution, NaOH- 1N NaOH solution*

Table D.2 Concentration of Selected Elements in the Silica Dissolution Study at 38°C Temperature Using ICP Method

| Element Concentration Detected | Fused Silica Exposure Combination | | | | |
|---|---|---|---|---|---|
| | Time Soaked (hrs) | NaOH+Si (38°C) | NaOH+Si+CH (38°C) | KAc +Si (38°C) | KAc +Si+CH (38°C) |
| Si | hrs | ppm | ppm | ppm | ppm |
| | 8.5 | 43.50 | 11.50 | 56.00 | 12.50 |
| | 26 | 183.50 | 14.00 | 50.50 | 7.50 |
| | 50 | 381.00 | 7.50 | 74.00 | 25.00 |
| | 192 | 2416.00 | 2416.00 | 105.50 | 26.00 |
| | 336 | 4274.00 | 916.50 | 116.50 | 29.00 |
| | 504 | 6551.00 | 1361.00 | 134.50 | 27.00 |
| | 672 | 6884.00 | 2378.50 | 135.00 | 27.50 |
| Ca | hrs | ppm | ppm | ppm | ppm |
| | 8.5 | -2.00 | 13.50 | 0.00 | 465.50 |
| | 26 | -4.00 | 11.50 | 1.00 | 277.00 |
| | 50 | -6.00 | 7.50 | 3.00 | 137.00 |
| | 192 | 43.00 | 43.00 | 0.00 | 406.50 |
| | 336 | 44.00 | 44.00 | 57.50 | 592.50 |
| | 504 | 44.00 | 43.00 | 50.50 | 1036.00 |
| | 672 | 44.00 | 44.00 | 54.50 | 1248.50 |
| Na | hrs | ppm | ppm | ppm | ppm |
| | 8.5 | 8398.00 | 6771.50 | 257.00 | 301.50 |
| | 26 | 7547.50 | 6986.50 | 186.00 | 191.50 |
| | 50 | 6431.00 | 4294.50 | 236.50 | 182.50 |
| | 192 | 6903.50 | 6903.50 | 294.00 | 231.00 |
| | 336 | 6337.50 | 6082.00 | 307.50 | 189.00 |
| | 504 | 6600.50 | 6094.00 | 248.00 | 326.00 |
| | 672 | 5617.50 | 5779.50 | 246.00 | 248.00 |
| K | hrs | ppm | ppm | ppm | ppm |
| | 8.5 | 813.50 | 271.50 | 121868.00 | 127133.00 |
| | 26 | 453.00 | 321.50 | 111801.00 | 109256.00 |
| | 50 | 311.00 | 465.50 | 121339.00 | 51711.50 |
| | 192 | 1633.00 | 1153.50 | 124389.50 | 46852.00 |
| | 336 | 117.50 | 117.50 | 59358.50 | 56488.00 |
| | 504 | 105.00 | 1363.00 | 52141.00 | 64944.50 |
| | 672 | 749.00 | 496.50 | 54329.50 | 46678.00 |

*Note: All concentrations are in 'ppm'- parts per million. Si- Fused Silica, CH-Calcium Hydroxide (Lime), KAc- 50% wt. potassium acetate deicer solution, NaOH- 1N NaOH solution*

# APPENDIX-E PH MEASUREMENT DATA

Table E.1 28 Day pH Measurements of Soak Solutions of Standard and Modified ASTM C 1567 Tests With Spratt Limestone and Fly Ash at 25% Dosage

| Fly Ash Type | %CaO | 28 day Readings | | | |
|---|---|---|---|---|---|
| | | KAc | | 1N NaOH | |
| | | pH | Temp.($^{o}$C) | pH | Temp.($^{o}$C) |
| LL1 | 1.3% | 14.2 | 21.6 | 13.6 | 21.4 |
| LL2 | 1.3% | 14.0 | 21.6 | 13.8 | 21.4 |
| LL4 | 7.3% | 14.1 | 21.4 | 13.9 | 21.2 |
| LL5 | 7.5% | 13.5 | 21.9 | 13.5 | 21.9 |
| IL1 | 10.3% | 14.8 | 21.6 | 13.6 | 21.7 |
| IL2 | 10.5% | 13.9 | 21.5 | 13.8 | 21.4 |
| IL3 | 11.6% | 14.2 | 21.8 | 13.5 | 21.8 |
| IL4 | 12.3% | 14.2 | 21.5 | 13.6 | 21.5 |
| IL6 | 18.9% | 14.2 | 21.5 | 13.7 | 21.4 |
| HL1 | 24.8% | 14.2 | 21.2 | 13.6 | 21.6 |
| HL2 | 27.5% | 14.0 | 21.8 | 13.5 | 21.8 |
| HL4 | 29.9% | 14.2 | 21.8 | 13.5 | 21.8 |

Table E.2 pH Measurements of Soak Solutions of Cement-Fly Ash Paste Samples at Different Ages

| | 15% | | 25% | | 35% | |
|---|---|---|---|---|---|---|
| | Cement- LOW LIME FLY ASH (LL3) | | | | | |
| Day | 1 N NaOH | KAc | 1 N NaOH | KAc | 1 N NaOH | KAc |
| 0 | 13.71 | 10.8 | 13.71 | 10.8 | 13.71 | 10.8 |
| 3 | 13.49 | 13.49 | 13.65 | 12.86 | 13.63 | 12.65 |
| 7 | 13.58 | 13.82 | 13.71 | 13.56 | 13.66 | 13.53 |
| 14 | 13.63 | 13.77 | 13.62 | 13.62 | 13.65 | 13.6 |
| 21 | 13.54 | 13.78 | 13.69 | 13.66 | 13.62 | 13.65 |
| Cement- INTERMEDIATE LIME FLY ASH (IL5) | | | | | | |
| Day | 1 N NaOH | KAc | 1 N NaOH | KAc | 1 N NaOH | KAc |
| 0 | 13.71 | 10.8 | 13.71 | 10.8 | 13.71 | 10.8 |
| 3 | 13.57 | 13.63 | 13.63 | 13.15 | 13.66 | 12.33 |
| 7 | 13.63 | 13.8 | 13.65 | 13.68 | 13.66 | 13.32 |
| 14 | 13.58 | 13.75 | 13.63 | 13.71 | 13.63 | 13.55 |
| 21 | 13.63 | 13.81 | 13.69 | 13.73 | 13.69 | 13.66 |
| Cement- HIGH LIME FLY ASH (HL3) | | | | | | |
| Day | 1 N NaOH | KAc | 1 N NaOH | KAc | 1 N NaOH | KAc |
| 0 | 13.71 | 10.8 | 13.71 | 10.8 | 13.71 | 10.8 |
| 3 | 13.63 | 13.67 | 13.66 | 13.52 | 13.58 | 13.05 |
| 7 | 13.66 | 13.91 | 13.66 | 13.83 | 13.66 | 13.67 |
| 14 | 13.59 | 13.88 | 13.62 | 13.82 | 13.65 | 13.7 |
| 21 | 13.64 | 13.93 | 13.67 | 13.85 | 13.68 | 13.75 |

Table E.3 pH Measurements of Soak Solutions of Cement-Slag Paste Samples and Control Cement Paste Samples at Different Ages

| | Cement- Slag | | Control (Cement Only) | |
|---|---|---|---|---|
| Day | 1 N NaOH | KAc | 1 N NaOH | KAc |
| 0 | 13.71 | 10.8 | 13.71 | 10.8 |
| 3 | 13.6 | 13.38 | 13.63 | 13.8 |
| 7 | 13.65 | 13.81 | 13.61 | 13.84 |
| 14 | 13.63 | 13.81 | 13.6 | 13.83 |
| 21 | 13.65 | 13.85 | 13.66 | 13.83 |

Table E4 pH Measurements of Soak Solutions of Modified ASTM C 1293 Test With Spratt, New Mexico and Dolomite Aggregate Concrete Prisms at 6 Month and 1 Year Test Age

| | SPRATT-1N NaOH | | | | | | SPRATT-KAc | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Fly Ash Dosage | Low Lime FA | | Inter. Lime FA | | High lime FA | | Low Lime FA | | Inter. Lime FA | | High lime FA | |
| | 180 d | 365 d | 180 d | 365 d | 180 d | 365 d | 180 d | 365 d | 180 d | 365 d | 180 d | 365 d |
| 25% | 13.56 | 13.61 | 13.66 | 13.61 | 13.43 | 13.65 | 14.16 | 14.22 | 14.4 | 14.48 | 14.39 | 14.44 |
| 35% | 13.48 | 13.58 | 13.6 | 13.64 | 13.45 | 13.58 | 14.22 | 14.19 | 14.25 | 14.28 | 14.33 | 14.35 |
| | NEW MEXICO-1N NaOH | | | | | | NEW MEXICO-KAc | | | | | |
| Fly Ash Dosage | Low Lime FA | | Inter. Lime FA | | High lime FA | | Low Lime FA | | Inter. Lime FA | | High lime FA | |
| | 180 d | 365 d | 180 d | 365 d | 180 d | 365 d | 180 d | 365 d | 180 d | 365 d | 180 d | 365 d |
| 25% | 13.54 | 13.58 | 13.64 | 13.38 | 13.6 | 13.42 | 14.26 | 14.23 | 14.38 | 14.18 | 14.15 | 14.24 |
| 35% | 13.51 | 13.62 | 13.62 | 13.49 | 13.6 | 13.53 | 14.15 | 14.16 | 14.23 | 14.14 | 14.23 | 14.25 |
| %SLAG | SPRATT-1N NaOH | | SPRATT-KAc | | New Mexico-1N NaOH | | New Mexico - KAc | | Dolomite-1N NaOH | | Dolomite-KAc | |
| | 180 d | 365 d | 180 d | 365 d | 180 d | 365 d | 180 d | 365 d | 180 d | 365 d | 180 d | 365 d |
| 40% | 13.54 | 13.54 | 14.38 | 14.38 | 13.72 | 13.54 | 14.08 | 14.32 | 13.71 | 13.49 | 14.48 | 14.33 |
| 50% | 13.51 | 13.52 | 14.33 | 14.4 | 13.71 | 13.64 | 14.22 | 14.4 | | | | |

# APPENDIX-F STATISTICAL ANALYSES

Table F.1 Matrix of LSD Test Results of Mortar Bar Expansions in 1N NaOH and KAc for Spratt Aggregate

| SP Limestone-1N NaOH | | | | | SP Limestone-Potassium Acetate | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Dosage | 0% | 15% | 25% | 35% | Dosage | 0% | 15% | 25% | 35% |
| 0% | | X | X | X | 0% | | X | X | X |
| 15% | X | | S | X | 15% | X | | S | X |
| 25% | X | S | | X | 25% | X | S | | X |
| 35% | X | X | X | | 35% | X | X | X | |
| Fly Ash Type | LL | IL | HL | Control | Fly Ash Type | LL | IL | HL | Control |
| LL | | X | X | X | LL | | S | X | X |
| IL | X | | X | X | IL | S | | X | X |
| HL | X | X | | X | HL | X | X | | X |
| Control | X | X | X | | Control | X | X | X | |

*(Note: 'X' represents a statistically significant difference between dosages or fly ash types and, '**S**' represents that the difference between the comparisons is similar or not significantly different)*

Table F.3 Matrix of LSD Test Results of Mortar Bar Expansions in 1N NaOH and KAc for NM Rhyolite Aggregate.

| NM Rhyolite-1N NaOH | | | | | NM Rhyolite-Potassium Acetate | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Dosage | 0% | 15% | 25% | 35% | Dosage | 0% | 15% | 25% | 35% |
| 0% | | X | X | X | 0% | | X | X | X |
| 15% | X | | X | X | 15% | X | | S | X |
| 25% | X | X | | X | 25% | X | S | | X |
| 35% | X | X | X | | 35% | X | X | X | |
| Fly Ash Type | LL | IL | HL | Control | Fly Ash Type | LL | IL | HL | Control |
| LL | | X | X | X | LL | | X | X | X |
| IL | X | | X | X | IL | X | | X | S |
| HL | X | X | | X | HL | X | X | | S |
| Control | X | X | X | | Control | X | S | S | |

*(Note: 'X' represents a statistically significant difference between dosages or fly ash types and, '**S**' represents that the difference between the comparisons is similar or not significantly different)*

Table F.3 Matrix of LSD Test Results of Mortar Bar Expansions in 1N NaOH and KAc for NC Argillite Aggregate

| NC Argillite-1N NaOH | | | | | NC Argillite-Potassium Acetate | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Dosage | 0% | 15% | 25% | 35% | Dosage | 0% | 15% | 25% | 35% |
| 0% | | X | X | X | 0% | | X | X | X |
| 15% | X | | S | X | 15% | X | | S | X |
| 25% | X | S | | X | 25% | X | S | | X |
| 35% | X | X | X | | 35% | X | X | X | |
| Fly Ash Type | LL | IL | HL | Control | Fly Ash Type | LL | IL | HL | Control |
| LL | | S | X | X | LL | | S | X | X |
| IL | S | | X | X | IL | S | | X | X |
| HL | X | X | | X | HL | X | X | | S |
| Control | X | X | X | | Control | X | X | S | |

*(Note: 'X' represents a statistically significant difference between dosages or fly ash types and, '**S**' represents that the difference between the comparisons is similar or not significantly different)*

Table F.4 Matrix of LSD Test Results of Mortar Bar Expansions in 1N NaOH and KAc for SD Quartzite Aggregate

| SD Quartzite-1N NaOH | | | | | SD Quartzite-Potassium Acetate | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Dosage | 0% | 15% | 25% | 35% | Dosage | 0% | 15% | 25% | 35% |
| 0% | | X | X | X | 0% | | X | X | X |
| 15% | X | | X | X | 15% | X | | X | X |
| 25% | X | X | | X | 25% | X | X | | X |
| 35% | X | X | X | | 35% | X | X | X | |
| Fly Ash Type | LL | IL | HL | Ctrl. | Fly Ash Type | LL | IL | HL | Ctrl |
| LL | | X | X | X | LL | | X | X | X |
| IL | X | | X | X | IL | X | | X | X |
| HL | X | X | | S | HL | X | X | | S |
| Control | X | X | S | | Control | X | X | S | |

*(Note: 'X' represents a statistically significant difference between dosages or fly ash types and, '**S**' represents that the difference between the comparisons is similar or not significantly different)*

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Table F.5 Matrix of LSD Test Results of Mortar Bar Expansions with Spratt limestone and 15 Fly Ashes of Varied Lime Contents at 25% Dosage in 1N NaOH Exposure.

| | | LL1 | LL2 | LL3 | LL4 | LL5 | IL1 | IL2 | IL3 | IL4 | IL5 | IL6 | HL1 | HL2 | HL3 | HL4 | Ctrl. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | %CaO | 1.27 | 1.34 | 3.35 | 7.31 | 7.49 | 10.33 | 10.45 | 10.56 | 12.25 | 15.63 | 18.94 | 22.85 | 27.47 | 27.5 | 29.85 | 0 |
| LL1 | 1.27 | | S | X | X | X | X | S | S | X | X | X | X | X | X | X | X |
| LL2 | 1.34 | S | | S | X | X | X | S | S | X | X | X | X | X | X | X | X |
| LL3 | 3.35 | X | S | | X | X | S | S | S | S | X | X | X | X | X | X | X |
| LL4 | 7.31 | X | X | X | | S | X | X | X | X | X | X | X | X | X | X | X |
| LL5 | 7.49 | X | X | X | S | | X | X | X | X | X | X | X | X | X | X | X |
| IL1 | 10.33 | X | X | S | X | X | | X | S | S | X | X | X | X | X | X | X |
| IL2 | 10.45 | S | S | S | X | X | X | | S | X | X | X | X | X | X | X | X |
| IL3 | 10.56 | S | S | S | X | X | S | S | | S | X | X | X | X | X | X | X |
| IL4 | 12.25 | X | X | S | X | X | S | X | S | | X | X | X | X | X | X | X |
| IL5 | 15.63 | X | X | X | X | X | X | X | X | X | | S | X | X | X | X | X |
| IL6 | 18.94 | X | X | X | X | X | X | X | X | X | S | | X | X | X | X | X |
| HL1 | 22.85 | X | X | X | X | X | X | X | X | X | X | X | | X | X | X | X |
| HL2 | 27.47 | X | X | X | X | X | X | X | X | X | X | X | X | | X | X | X |
| HL3 | 27.5 | X | X | X | X | X | X | X | X | X | X | X | X | X | | X | S |
| HL4 | 29.85 | X | X | X | X | X | X | X | X | X | X | X | X | X | X | | S |
| Ctrl. | | X | X | X | X | X | X | X | X | X | X | X | X | X | S | S | |

*(Note: 'X' represents a statistically significant difference between the fly ash types and, '**S**' represents that the difference between the fly ash types compared is similar or not significantly different.)*

Table F.6 Matrix of LSD Test Results of Mortar Bar Expansions with Spratt limestone and 15 Fly Ashes of Varied Lime Contents at 25% Dosage in KAc Exposure.

| | | LL1 | LL2 | LL3 | LL4 | LL5 | IL1 | IL2 | IL3 | IL4 | IL5 | IL6 | HL1 | HL2 | HL3 | HL4 | Ctrl. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | %CaO | 1.27 | 1.34 | 3.35 | 7.31 | 7.49 | 10.33 | 10.45 | 10.56 | 12.25 | 15.63 | 18.94 | 22.85 | 27.47 | 27.5 | 29.85 | 0 |
| LL1 | 1.27 | | S | S | X | S | S | S | S | S | X | S | X | X | X | X | X |
| LL2 | 1.34 | S | | S | S | S | S | S | S | S | X | X | X | X | X | X | X |
| LL3 | 3.35 | S | S | | X | S | S | S | S | S | X | S | X | X | X | X | X |
| LL4 | 7.31 | X | S | X | | S | X | S | S | S | X | X | X | X | X | X | X |
| LL5 | 7.49 | S | S | S | S | | S | S | S | S | X | X | X | X | X | X | X |
| IL1 | 10.33 | S | S | S | X | S | | S | S | S | X | S | X | X | X | X | X |
| IL2 | 10.45 | S | S | S | S | S | S | | S | S | X | X | X | X | X | X | X |
| IL3 | 10.56 | S | S | S | S | S | S | X | | S | X | X | X | X | X | X | X |
| IL4 | 12.25 | S | S | S | S | S | S | S | S | | X | X | X | X | X | X | X |
| IL5 | 15.63 | X | X | X | X | X | X | X | X | X | | X | X | X | X | X | X |
| IL6 | 18.94 | S | X | S | X | X | S | X | X | X | X | | X | X | X | X | X |
| HL1 | 22.85 | X | X | X | X | X | X | X | X | X | X | X | | X | X | X | X |
| HL2 | 27.47 | X | X | X | X | X | X | X | X | X | X | X | X | | X | X | X |
| HL3 | 27.5 | X | X | X | X | X | X | X | X | X | X | X | X | X | | S | X |
| HL4 | 29.85 | X | X | X | X | X | X | X | X | X | X | X | X | X | S | | X |
| Ctrl. | | X | X | X | X | X | X | X | X | X | X | X | X | X | X | X | |

*(Note: 'X' represents a statistically significant difference between the fly ash types and,* ***'S'*** *represents that the difference between the fly ash types compared is similar or not significantly different)*

Table F.7 Matrix of Results of LSD Test to Determine the Differences in Mortar Bar Expansions due to (a) Slag Dosage and (b)Aggregate Type in 1N NaOH and KAc Exposure.

| 1N NaOH | | | | Pot. Acetate | | | |
|---|---|---|---|---|---|---|---|
| Dosage | 0% | 40% | 50% | Dosage | 0% | 40% | 50% |
| 0% | | X | X | 0% | | X | X |
| 40% | X | | *S* | 40% | X | | X |
| 50% | X | *S* | | 50% | X | X | |

| 1N NaOH | | | | | | Potassium Acetate | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Agg. Type | SP | NM | NC | SD | IL | Agg. Type | SP | NM | NC | SD | IL |
| SP | | X | *S* | *S* | *S* | SP | | X | *S* | *S* | X |
| NM | X | | X | X | X | NM | X | | X | X | X |
| NC | *S* | X | | *S* | *S* | NC | X | X | | *S* | *S* |
| SD | *S* | X | *S* | | *S* | SD | *S* | X | *S* | | X |
| IL | *S* | X | *S* | *S* | | IL | X | X | *S* | X | |

*(Note: 'X' represents a statistically significant difference between slag dosages or aggregate types and,* ***'S'*** *represents that the difference between the comparisons is similar or not significantly different.)*

Table F.8 Matrix of Results of LSD Test to Determine the Differences in Concrete Prism Expansions due to Fly Ash Dosage and Fly Ash Type in 1N NaOH and KAc Exposure for SP Limestone Aggregate.

| 1N NaOH | | | | Pot. Acetate | | | |
|---|---|---|---|---|---|---|---|
| Dosage | 0% | 25% | 35% | Dosage | 0% | 25% | 35% |
| 0% | | X | X | 0% | | *S* | X |
| 25% | X | | X | 25% | *S* | | *S* |
| 35% | X | X | | 35% | X | *S* | |

| 1N NaOH | | | | | Potassium Acetate | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Fly Ash Type | LL | IL | HL | Control | Fly Ash Type | LL | IL | HL | Control |
| LL | | X | X | X | LL | | *S* | X | X |
| IL | X | | X | X | IL | *S* | | X | X |
| HL | X | X | | X | HL | X | X | | X |
| Control | X | X | X | | Control | X | X | X | |

Table F.9 Matrix of Results of LSD Test to Determine the Differences in Concrete Prism Expansions due to Fly Ash Dosage and Fly Ash Type in 1N NaOH and KAc Exposure for NM Rhyolite Aggregate.

| 1N NaOH | | | | Pot. Acetate | | | |
|---|---|---|---|---|---|---|---|
| Dosage | 0% | 25% | 35% | Dosage | 0% | 25% | 35% |
| 0% | | X | X | 0% | | X | *S* |
| 25% | X | | *S* | 25% | X | | X |
| 35% | X | *S* | | 35% | *S* | X | |

| 1N NaOH | | | | | Potassium Acetate | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Fly Ash Type | LL | IL | HL | Control | Fly Ash Type | LL | IL | HL | Control |
| LL | | X | X | X | LL | | *S* | X | *S* |
| IL | X | | X | X | IL | *S* | | X | *S* |
| HL | X | X | | X | HL | X | X | | X |
| Control | X | X | X | | Control | *S* | *S* | X | |

Table F.10 Matrix of Results of LSD Test to Determine the Differences in Concrete Prism Expansions due to Slag Dosage and Aggregate Type in 1N NaOH and KAc Exposure

| 1N NaOH | | | | Pot. Acetate | | | |
|---|---|---|---|---|---|---|---|
| Dosage | 0% | 40% | 50% | Dosage | 0% | 40% | 50% |
| 0% | | X | X | 0% | | *S* | *S* |
| 40% | X | | *S* | 40% | *S* | | *S* |
| 50% | X | *S* | | 50% | *S* | *S* | |
| 1N NaOH | | | | Pot. Acetate | | | |
| Agg. Type | NM | SP | IL | Agg. Type | NM | SP | IL |
| NM | | *S* | X | NM | | X | X |
| SP | *S* | | *S* | SP | X | | X |
| IL | X | *S* | | IL | X | X | |

*(Note: 'X' represents a statistically significant difference between slag dosages or aggregate types and,* ***'S'*** *represents that the difference between the comparisons is similar or not significantly different.)*

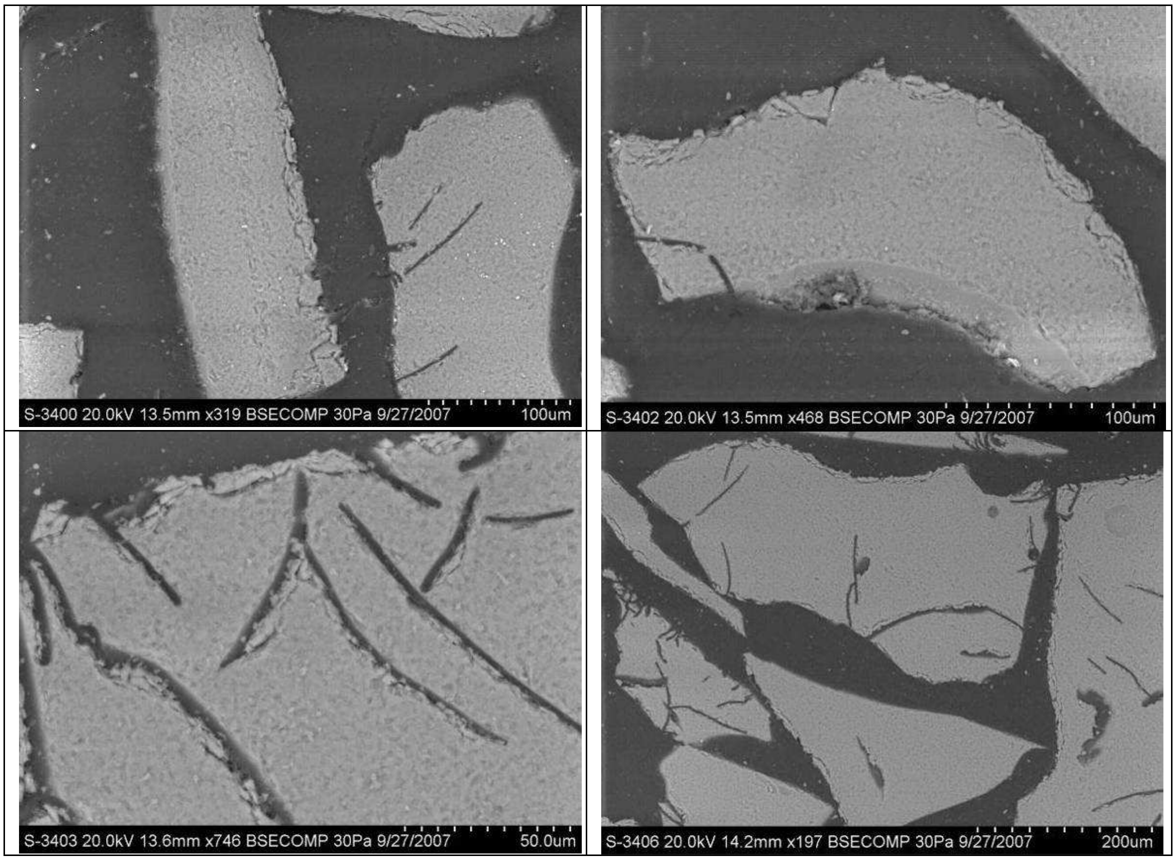


Figure G.1 SEM Images of Fused Silica Particles Exposed to 1N NaOH for 90 Days

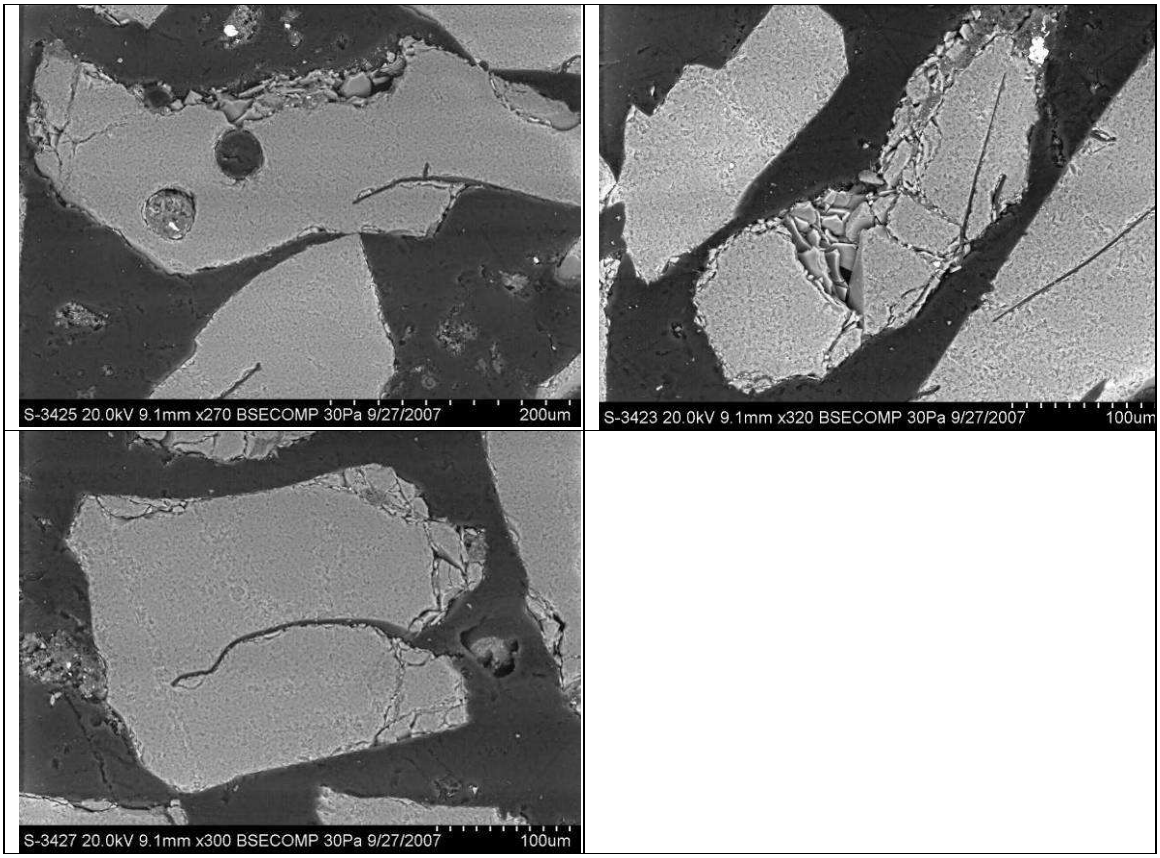


Figure G.2 SEM Images of Fused Silica Particles Exposed to 1N NaOH in Presence of Calcium Hydroxide for 90 Days

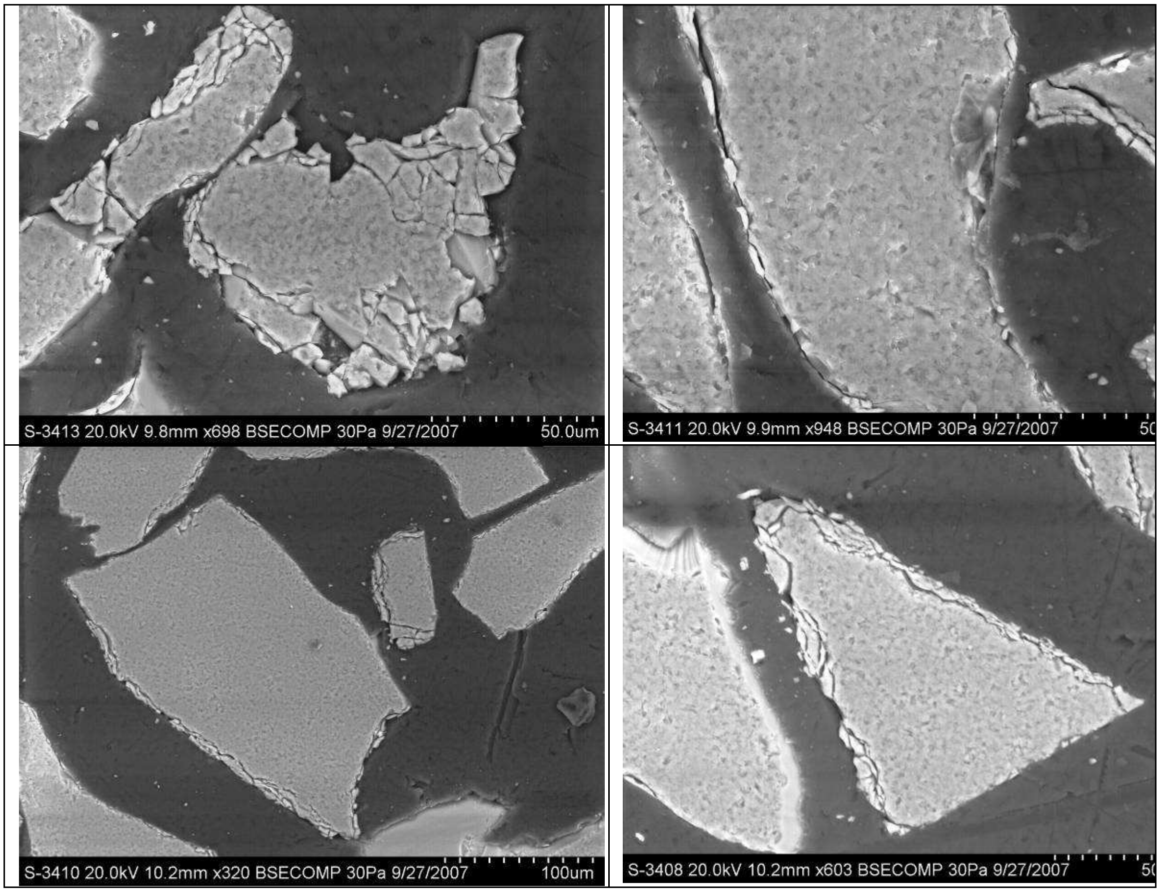


Figure G.3 SEM Images of Fused Silica Particles Exposed to Potassium Acetate Deicer Solution for 90 Days

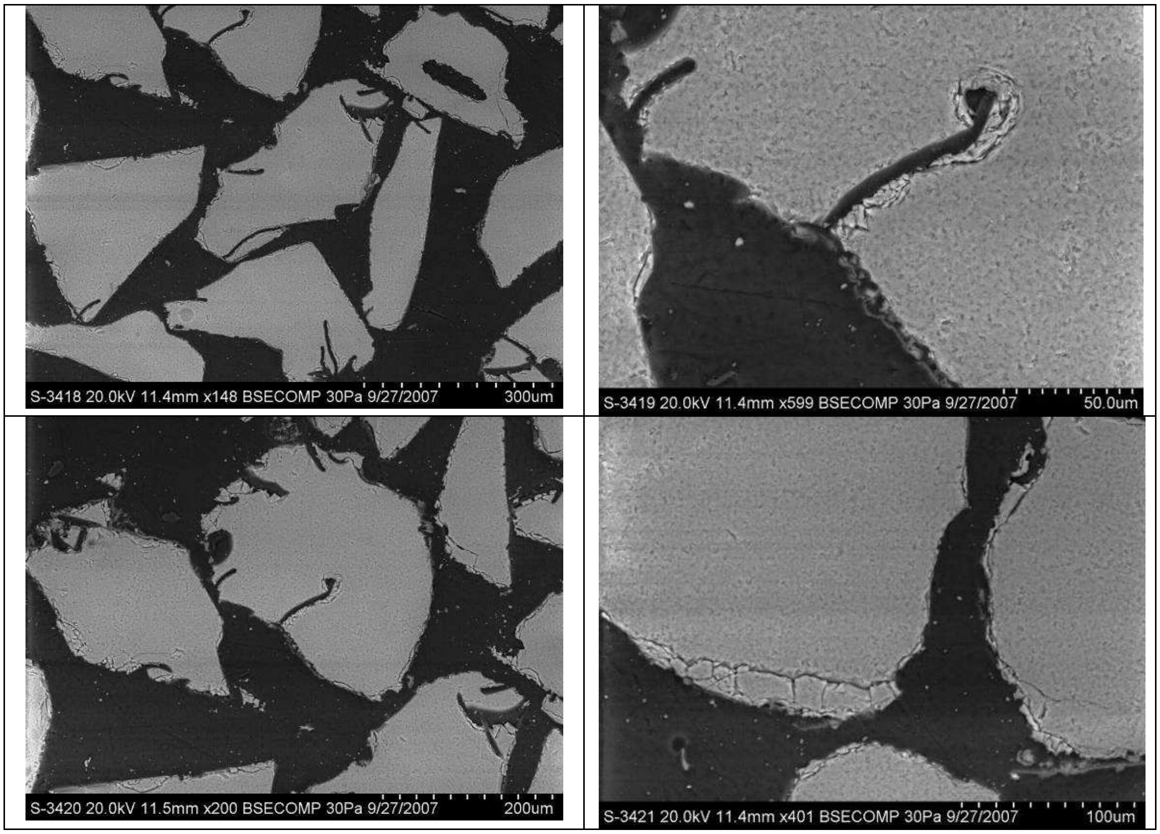


Figure G.4 SEM Images of Fused Silica Particles Exposed to Potassium Acetate Deicer Solution in Presence of Calcium Hydroxide for 90 Days

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